ADVANCES IN OIL SANDS TAILINGS RESEARCH

Prepared by
The Fine Tailings Fundamentals Consortium

Suncor Inc.
Oil Sands Group

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Securing Canada's Energy Future

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FOREWORD

The Problem

It took about a year from the commencement of commercial extraction of bitumen from oil sand in 1967 to fully recognize that the tailings pond was filling rapidly with a slowly dewatering suspension of fine mineral particles, now called "fine tailings." It was necessary to provide storage for this unanticipated fluid volume, which accumulated at a rate of more than 20% of the volume of oil sand excavated from the mine. Provision for containment of this fluid layer represents a direct incremental operating cost and continues to be the source of major logistical planning headaches. Additionally, it is necessary to accommodate these large volumes of fluid fine tailings in the development of an environmentally acceptable reclamation plan.

Research work began almost immediately to develop methods to eliminate or reduce the rate of accumulation of fine tailings. This effort continued for two decades without finding commercially and environmentally acceptable solutions. By 1988, the Athabasca oil sands were providing more than 15% of Canada's oil requirements. There were known to be vast reserves in the Athabasca deposit which could replace declining conventional reserves. Lack of commercial demonstration of progress in resolving fine tailings disposal issues was of concern to the operating companies, regulatory agencies, the public, and potential investors in further development projects.

The Collaboration

In 1989 a rather unique step was taken in the oil industry through the formation of the Fine Tailings Fundamentals Consortium. The initial mandate of the Consortium was to provide sufficient basic scientific understanding of fine tailings to form the basis for engineering solutions. The formal participating organizations eventually grew to include Alberta Energy, Alberta Research Council, Alberta Oil Sands Technology and Research Authority (AOSTRA), Environment Canada, Canada Centre for Mineral and Energy Technology (CANMET), National Research Council, Other Six Lease Owners (OSLO), Suncor, and Syncrude Canada Ltd, with AOSTRA acting as the operator for legal and business purposes. A Management Committee consisting of one voting member from each participant was formed to provide general policy direction and manage the Consortium's activities. A Technical Committee was also formed to develop a prioritized R&D plan and to implement the plan following approval by the Management Committee. The Technical Committee consisted of technical experts from very diverse backgrounds including colloidal scientists, clay scientists, chemists, microscopists, geotechnical engineers, process engineers, geologists, biologists, toxicologists, materials handling practitioners, mine logistics planners, and environmental scientists. They represented each participating organization, observers from various government groups, universities, members of organizations under contract to the Consortium, and outside experts. One vote from each formally participating organization was allowed when decisions were required.

Although originally intended just to provide basic scientific data, the Consortium developed into a catalyst for a powerful collaboration which significantly leveraged initial investment by participants. How this worked is shown in the attached Figure. Alberta Energy, AOSTRA, Environment Canada, and CANMET jointly contributed an average of $450K per year for the five-year life of the Consortium. Contributions made "in kind" from the research community actually generated an average of $1.25 million per year worth of research effort. This work was focused at the laboratory scale. At the same time, the operating companies contributed additional laboratory and relatively expensive pilot plant scale testing, the direction and scope for which was directly influenced by the collaboration provided by the Consortium. The expenditure for this work averaged $2.6 million per year, bringing total expenditure by the collaboration to $3.85 million average per year. The leverage on the original government cash investment works out to a multiplier of more than 8 to 1.

The Success

Has the Consortium been successful? Full capture of the Consortium's influence will not be confirmed in commercial operation for several more years. This is driven by the magnitude of the problem, technical and commercial risk, and implementation opportunity. One objective, enhancement of reclamation security, cannot really be evaluated for at least a quarter century.

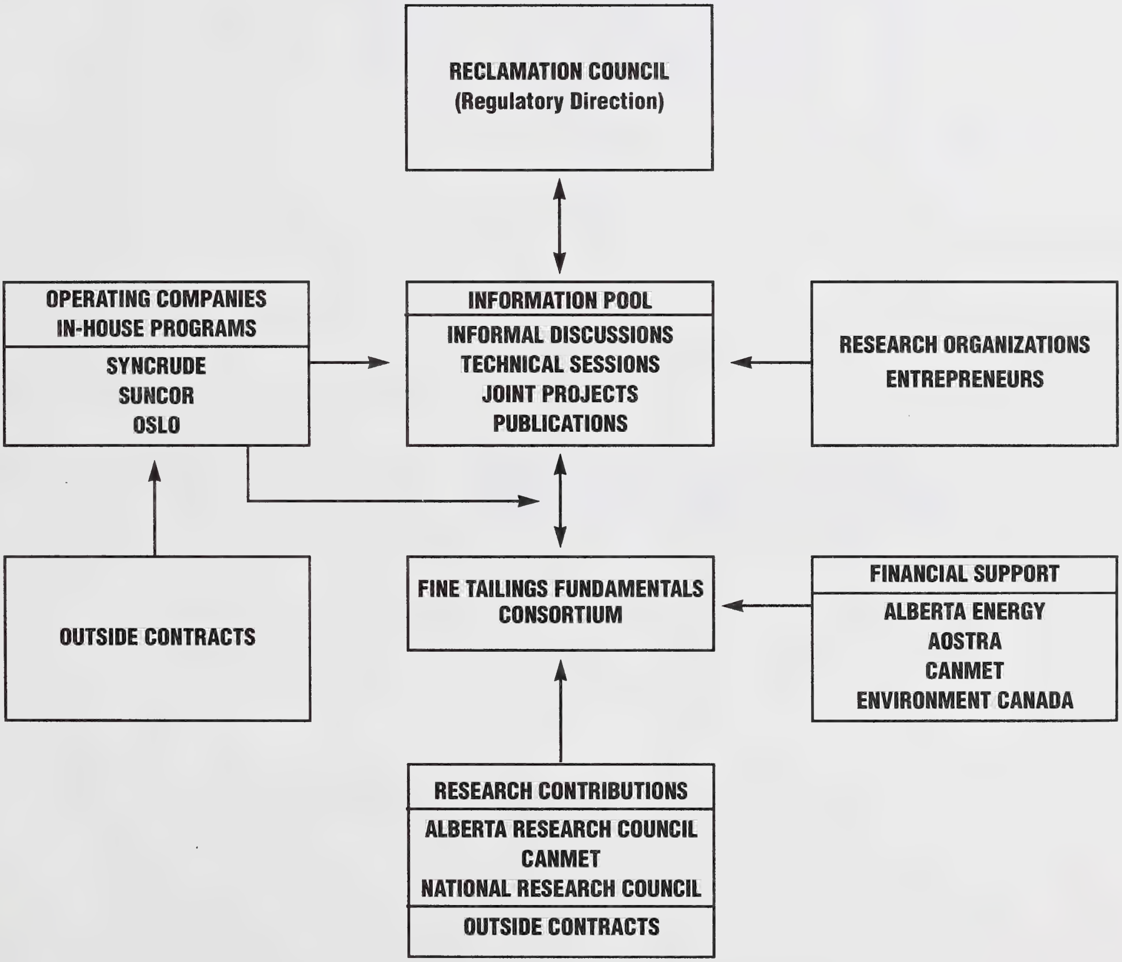
However, a preliminary assessment is possible. The most direct evidence concerns the response of the operating companies. New alternatives have been developed which are being investigated in detail through tailings planning activities and economic analysis within the operating companies. An additional $10 million will be spent in 1995 on large scale pilot plants investigating methods which resulted from the Consortium catalyzed collaboration.

A second line of evidence of success is the advance in scientific knowledge of fine tailings and in the ability to modify its behavior to improve its properties. This information is contained in this Final Report, consisting of four volumes. The intent is to provide a state of the art reference of important issues and to direct the interested reader to additional sources of information. The first volume describes the nature and properties of fine tailings generated by the Clark Hot Water Extraction (CHWE) process, the process used in both commercial operations. Volume II develops the CHWE tailings environmental issues and describes the water capped lake reclamation plans. Volume III explores methods to reincorporate CHWE fluid fine tailings back in stable soil deposits leading to increased use of dry landscape reclamation alternatives. Volume IV looks at the nature of fine tailings and reclamation opportunities resulting from alternate bitumen extraction processes.

The Future

The Fine Tailings Fundamentals Consortium has completed its mandate with the issuance of this Final Report following five years of operation. There will be issues to resolve as the operating companies progress toward implementation, and a team of experts fully familiar with oil sand tailings specific problems has been developed to address these issues. It is anticipated that the essential work will continue within the Canadian Oil Sands Network for Research and Development (CONRAD). For further information about CONRAD, contact Alberta Department of Energy, Oil Sands and Research Division by telephone at 403-427-7623.

TAILINGS RECLAMATION R&D COLLABORATION



VOLUME I

CLARK HOT WATER
EXTRACTION
FINE TAILINGS

CONTENTS

1.0 INTRODUCTION AND BACKGROUND

1.1 History of Oil Sands Development

Historically, oil sands have been known since the first explorers ventured into the Fort McMurray area in the 1760s. They discovered a black tar pitch oozing from the sand banks along the Athabasca River. Natives living in the area used this pitch to construct and repair their canoes, and talked of the extensive range of the deposit along the river bank. Little did they know that this deposit would be one of the world's largest reserves of hydrocarbons.

Beginning in the early 1920s, serious efforts were undertaken by the Alberta Government and various entrepreneurs to develop the commercial potential of these vast oil sands deposits [1]. Between 1920 and 1950, two major extraction and refining plants were built. The Abasand plant, located about 4 km west of Fort McMurray was constructed in the early 1930s to process approximately 250 tons/day of oil sands, using hot water and caustic for conditioning the oil sand and the application of raw distillate to dilute the bitumen and improve product quality. The plant operated intermittently from 1936 to 1938, and was then rebuilt over the next three years, with financial assistance from the wartime Federal Government, to improve its capacity and refine the bitumen into kerosene, gasoline, and fuel oil. In 1943, this plant was again remodeled and produced bitumen at rates of 2000 – 4000 bbl/month. After a fourth expansion, the plant caught fire on June 16,1945 and was never rebuilt.

In parallel with the Abasand project, the International Bitumen Company was constructing and operating a extraction and refining operation producing fuel oil, roofing tar, and asphalt for road paving. This tiny operation was unsuccessful in raising sufficient capital to expand during the 1930s and was shut down by 1939. However, the wartime needs for fuel and the arrival of a new financier, Lloyd Champion, resulted in the construction of a much larger plant at the Bitumont site which included mining, extraction, and refining. After a brief operating period in 1949 the plant, owned by Oil Sands Ltd., was shut down as it was unable to sustain continued operations. However, the property owned by Lloyd Champion and Oil Sands Ltd. included the lease upon which Suncor now stands, and this development was the forerunner of Great Canadian Oil Sands, now Suncor.

Throughout the development of the oil sands, the Alberta Research Council has played a key role, particularly with the work of Dr. Karl Clark. Beginning in the early 1920s, Dr. Clark experimented with the hot water process and in 1924 operated a pilot plant to demonstrate the larger scale extraction of bitumen from oil sands. Numerous modifications were made to this plant through the 1920s and the operating data was used to make projections as to the necessary scale of operations required for a commercial venture. Also, Dr. Clark's team investigated numerous uses for oil sand and bitumen products, including its use for road asphalt and roofing tar. However, his main contribution to the development of the oil sands was the large number of scientific publications and technical information which formed the basis of the large scale commercial developments that exist today.

Currently, about one-third of Canada's supply of oil comes from the oil sands deposits. Production of bitumen from surface mines operated by Syncrude and Suncor totals 500,000 tonnes daily and the shipments of light sweet synthetic crude oil totals 42,000 m^3/d (260,000 bbl/d), or approximately 15% of Canada's conventional oil. *In situ* production of bitumen from the deep oil sands deposits results in an additional 16,000 m^3/d (100,000 bbl/d). These volumes contribute to the overall 58,000 m^3/d or 360,000 bbl/d out of Canada's total oil production of 2.4M bbl/day. As conventional oil reserves decline, this resource will continue to become more significant in Canada's overall energy picture.

1.2 Oil Sands

The Athabasca deposit is the largest of the four oil sands areas in Alberta (Athabasca, Cold Lake, Wabasca, Peace River) with estimated in-place reserves of 869 billion barrels of bitumen [2]. The near-surface occurrence of the oil sands in the Fort McMurray area has allowed for the deposit to be recovered using surface mining techniques.

Essentially all of the bitumen reserves in the Athabasca deposit are contained within the Lower Cretaceous McMurray Formation. This formation comprises 30 to 90 meters of sediments deposited in different depositional environments. In the two surface mining areas (Suncor Mine and Syncrude Mine) sediments have been deposited in fluvial, estuarine, and marine environments. Because the McMurray Formation sediments have experienced very little post-depositional diagenetic effects the distribution of bitumen saturated sediments is related to the primary paleogeographic distribution of the sediments. In brief, the bitumen distribution is predominantly facies controlled [3].

The sediments generally consist of uncemented quartzose sands and associated finer-grained lithologies. Locally, these clastic sediments are bitumen-bearing over the entire formation thickness. More commonly, the bitumen-bearing sediments are interbedded with barren units of water sands and clay-rich fine-grained sediments. Average bitumen saturation within ore bodies is greater than 10 weight percent.

The bitumen impregnated sands consist of sand grains with silt, clay, water, and bitumen filling the intergranular spaces. These silt and clay particles together with those from the barren interbeds become dispersed during the Clark Hot Water Extraction process and largely accumulate as fine tails deposits in the tailings settling basins.

The clay minerals within the McMurray Formation are predominantly kaolinite and illite. Minor and trace amounts of smectite, chlorite, vermiculite, and mixed-layer clays are also present. In general, there is more smectite and mixed-layer clays in the marine sediments than in the estuarine and fluvial sediments. Vermiculite is most prevalent in the fluvial sediments [4].

The clays in the fine tails reflect those in the oil sand ore and have not been altered mineralogically during the bitumen extraction process. Typically, kaolinite comprises 50 to 60% and illite 30 to 50% of the clay minerals in fine tails.

1.3 Extraction Process

A number of processes have been considered for extracting the bitumen from the oil sands. A brief review of some of these processes is given by Fuhr *et al.* [5]. A schematic flow diagram of the oil sands process, from open pit mine to final product is shown in Figure 1-1.

The extraction process used by the two oil sands plants and referred to as a hot water process is based on the pioneer work of Dr. Karl Clark, who used a combination of hot water, steam, and caustic to separate the bitumen from the oil sand. These concepts were pilot tested in the early 1960s, first by a group led by Cities Service Athabasca, and later by Great Canadian Oil Sands, the precursor of Suncor. The major process steps which were developed in these pilot operations have remained essentially unchanged and form the basis for today's commercial operations.

In today's commercial plants, oil sand is mined at Suncor with truck and shovel operations, and at Syncrude using a combination of draglines/bucketwheels and truck and shovel methods. Delivery of the oil sand to the plant is accomplished with conveyors. From the conveyor discharge, the oil sand is digested and conditioned in large tumblers with the addition of hot water, caustic soda (NaOH), and steam. In the resulting slurry, the bitumen separates from the sand and is aerated, resulting in a froth that will readily separate from the slurry in the next separation stage. Rocks and balled clay-rich wastes which have been carried through the digestion stage are removed by vibrating screens at the tumbler discharge. Slurry produced in this stage is diluted with additional quantities of hot water and pumped to the separation vessels.

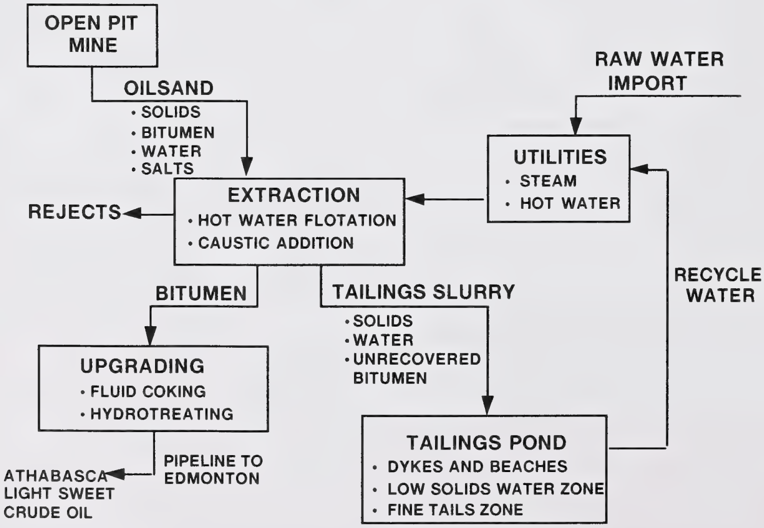


FIGURE 1-1. Generalized scheme for processing oil sands at Syncrude and Suncor.

Bitumen is separated as a froth which floats to the surface in large vessels known as primary separation vessels. The coarse sand settles to the bottom and is removed. A portion of the slurry, known as middlings, is removed from the central portion of the vessel, and is further processed to recover the fine oil droplets which remain unfloated in the primary separation process. At Suncor, this stream is processed in conventional flotation cells, and the small quantity of recovered oil is added to the product from the primary settling vessels. At Syncrude, the tailings and middlings streams from the primary separation vessel are combined and further processed in large, deep cone vessels where additional oil is recovered from both the middlings and coarse tailings streams. This bitumen froth is also added to the froth from the primary settling vessels. Both Suncor and Syncrude generate bitumen froth products which contain significant quantities of water and fine solids, mainly clays which must be removed prior to the upgrading process where bitumen is converted into a light synthetic crude oil.

1.4 Tailings Disposal Method

Tailings from the primary and secondary separation vessels are mainly solids and water with small residual quantities of bitumen. In addition, froth treatment tailings are pumped to the tailings ponds. In summary, the tailings are a warm aqueous suspension of sand, silt, clay residual bitumen, and naphtha at a pH between 8 and 9.

Other than the screen oversize or reject taken from the tumblers which is placed back in the mined-out pit, all the other tailings are pumped to large tailings ponds, where the coarse solids settle out to form dykes and beaches while much of the fines and residual bitumen are carried into the pond as a thin slurry stream (about 8 wt% solids). Once the stream slows down, sedimentation of the clastic particles begins. Stokian and hindered settling take place leaving a largely clarified water layer at the surface of the pond. The clarified water is recovered and recycled as extraction process water. A cross section of the oil sands tailings settling basin is shown schematically in Figure 1-2.

As mentioned above, the feed to the extraction plant also contains significant quantities of fine-grained materials, which remain in the slurry and are carried with the sand to the tailings pond. These fine-grained clastic materials (silts and clays) and the residual bitumen that sinks in the pond form an accreting layer which consolidates very slowly, or not at all. The resulting deposit of fine-grained sediment, water, and bitumen are known as "fine tailings" or "fine tails."

Containment of the entire water system within the operating process is required as part of the operating license agreements between the Provincial Government and the two commercial plants (Suncor has been permitted to discharge a small amount of water, but this is not significant in the overall water utilization balance). Since discharge into the environment is not permitted, the main loss of water is to the voids in the tailings sand as the beaches are formed. Losses to evaporation are small, and thus only relatively minor quantities of water are lost. Fresh water required for make-up is small in comparison with the water which is recycled through the process. Without the benefit of addition of large quantities of clean dilution water, recycle of water between the process and the tailings ponds in this closed loop has resulted in accumulation of fine clays and any dissolved inorganic ions in the tailings pond water system. This process has been on-going since the start up of both plants. Currently, millions of cubic meters of fine tailings are present in the tailings ponds, presenting a major environmental concern and necessitating development of environmentally acceptable disposal methods for the long term.

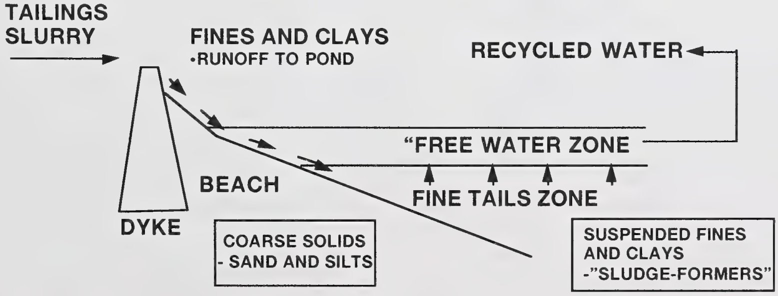


FIGURE 1-2. Cross section of oil sands tailings settling basin.

Over time, three zones have developed in the tailings ponds [6]. On top is about three meters of clear water which is continuously pumped back into the plant for reuse. Under the top layer is a transition zone of water and settling clay particles, about one meter thick. At the bottom is the fine tailings zone, a layer of clays, fine sand, bitumen, and water which increases in density with depth and is over 40 meters thick in some areas. Syncrude's fine tailings are about 85% water, 13% clays, and 2% bitumen, and are considered acutely toxic because fish cannot live in them. In addition to the high concentration of solids, the water in the fine tailings contains a number of organic compounds such as naphthenic acids which are derived from the bitumen. Most of these organics are believed to be released from the oil sand during extraction, but can also be naturally occurring when oil is eroded by local streams. These compounds also contribute to the toxicity of the fine tailings.

It is generally thought that the extremely slow consolidation of the fine tails is related to the dispersed nature of the fine and ultrafine particles as well as the ionic chemistry of the process water.

1.5 Fine Tails Volumes

Oil sands processing and the disposal of the tailings has been on-going since 1968 at Suncor and since 1978 at Syncrude. The cumulative ore volumes processed and the cumulative fine tails volumes generated are shown in Figures 1-3 and 1-4 for the Suncor and Syncrude operations, respectively [7].

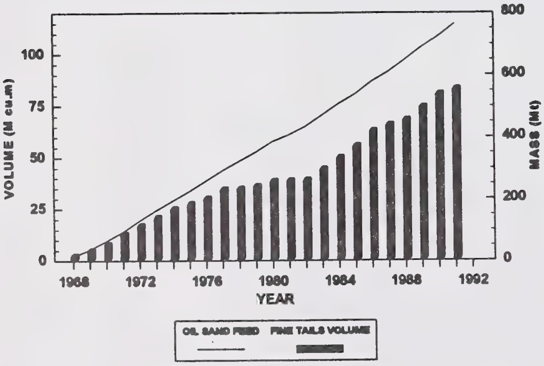


FIGURE 1-3. Cumulative mass of oil sand feed and fine tails volume in tailings ponds at Suncor: 1968 – 1991.

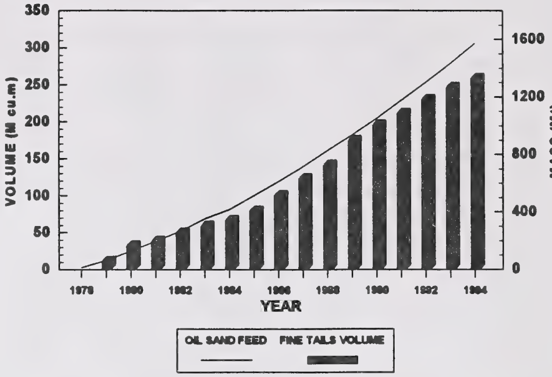


FIGURE 1-4. Cumulative mass of oil sand feed and fine tails volume in tailings ponds at Syncrude: 1978 – 1994.

As can be seen in the above figures large volumes of fine tails have accumulated in tailings settling basins to date. Considerable effort has been expended in the past decade by the oil sands mining and processing operators as well as various government and research institutions to identify suitable technologies to reclaim existing fine tails inventories and reduce the future volumes of fine tails produced.

1.6 Fine Tails — The Challenge

Although less than 10% of the total bitumen reserves in the Athabasca deposit are amenable to surface mining, this method of exploitation of the oil sands is currently the most economic and may remain so for some time. The large volumes of fine tails generated through surface mining and water-based processing of the oil sands provide a major challenge. Further development of the surface mineable portion of the oil sands could depend on identifying economically and environmentally viable solutions.

Storage and disposal of large volumes of fine tails remains one of the major challenges associated with the Clark Hot Water Extraction process. For this reason, considerable research has been conducted in recent years in an effort to identify ways of reducing or eliminating the environmental concerns associated with reclamation of these wastes.

1.7 References

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2.0 EXTRACTION FACTORS

2.1 Introduction

This chapter describes what is known about the effect of extraction factors on the Clark process. Extraction factors that are important include the ore, the water and process parameters such as the amount of sodium hydroxide used, the temperature and the amount of mechanical energy. These factors influence recovery from the process and also affect the properties of the tailings which include settling rate, toxicity, final settled volume and ease of compaction for ultimate disposal.

2.2 Effect of Ore Properties on the Process

The primary recovery obtainable using the Clark process for several representative ores has been shown [1] to be a function of the amount of sodium hydroxide used. There is an optimum amount of sodium hydroxide for each ore that produces maximum primary recovery. The amount can vary from none for rich ores to as much as 0.10 wt% for very low grade ores.

The fines content (defined as the fraction of solid particles of <44 μm size) of oil sands, determines the amount of sodium hydroxide needed to maximize primary recovery [1]. In general, the greater the fines content, the greater the requirement for sodium hydroxide with a linear relationship existing and an equation available for calculating the amount needed from the fines content of the ore.

Even with the adjustment made to optimize the amount of sodium hydroxide, high fines ores give poorer primary recoveries than low fines ores. Data has been reported and plotted [1] for several ores and the plot can be used to predict primary recovery for a given oil sand from the fines content.

The effect of the ore on the final settled volume of mature fine tails was investigated by the Fine Tails Consortium. The results and data have been reported [2,3].

Experimentally, some typical Syncrude oil sands were processed using a laboratory scale version of the Clark process [1]. The fine tailings from the experiments were allowed to settle for one year in graduated cylinders. Settling had almost ceased at this time and the volumes of sediment were determined and are referred to as final sediment volumes. There was a large variation in these volumes.

The oil sands used in the study were analyzed extensively to measure properties that it was thought might play some role in determining final sediment volume. The measurements included fines content (Microtrac), bitumen content, exchangeable cations, clay content (X-ray diffraction), methylene blue uptake, metals in ash, and amorphous iron and calcium.

Statistically, linear models of the forms shown in Eqs. (1) and (2) were tested.

$$(1)\quad Y = \beta_o + \Sigma\left(\beta_i x_i\right)$$

$$(2)\quad Y = \Sigma\left(\beta_i x_i\right)$$

The models were tested using from 1 to 5 values of x but it was found that only the one variable models were statistically significant. The use of more than one variable in the model increased the value of R^2, but the equations which resulted were not statistically significant.

The importance of the measured ore property in contributing to the final settled volume was examined using only the one variable models. Eq. (1) was first tested, if this model failed the statistical tests, then Eq. (2) was used.

Ore properties which best correlated with the final sediment volume were properties which in some way were a measure of the clay content of the ore. Methylene blue uptake gave an R^2 of 0.86 and exchangeable potassium gave an R^2 of 0.71. Methylene blue uptake has been related [4] to clay content as has exchangeable potassium [5]. The indication is that methylene blue uptake is the best clay measurement method for purposes of estimating final sediment volume.

The data also showed that the <44 μm fines value, as measured by Microtrac, is a poor predictor of final sediment volume. The R^2 was 0.29 for the <44 μm fraction and gradually increased to 0.71 for the <2.8 μm fraction.

Another study aimed at comparing fine tails from estuarine and marine oil sands suggests that geochemistry of the ore plays a major role in the settling rate of the fines, particularly the clay size fines [3]. The chemistry of the tailings water relates in part to the mineralogy of the oil sand as well as the interstitial water (formation water). In general, the calcium and magnesium in the marine ore is higher than in the estuarine ore. The higher amounts of soluble divalent ions in the marine ore appears to contribute to enhanced flocculation of clay fines which may result in increased retention of fine tails "formers" on the tailings beaches and faster settling of fine tails in tailings ponds.

2.3 Effect of Water Chemistry on the Process

There are a variety of water sources that either have been used or have been proposed for use in processing oil sands.

The list includes river water, recycle pond water, dionized water, city water, and pond water or river water after treatment with sulfuric acid and lime [6]. River water, treated pond water, and treated river water have been suggested [6] for use in processing oil sands by the OHWE process. The effect of water chemistry on the OHWE process is discussed in Volume IV. Recycle pond water is used commercially as both slurry water and flood water to process oil sands by the Clark process. Deionized water has been used extensively in laboratory studies on the Clark process, and City of Edmonton water has been used almost exclusively in pilot scale work on the Clark process at Syncrude. The effect of water type on recovery and tailings behavior on the Clark process is of interest and has been investigated.

Analytical data comparing recycle pond water to City of Edmonton water are recorded in Table 2-1.

TABLE 2-1.

Species	Pond water	City water
Cl^- (ppm)	280	3
SO_4^{2+} (ppm)	259	84
HCO_{3-} (meq/L)	14.0	0.8
Mg^{2+} (ppm)	4.6	11
Ca^{2+} (ppm)	9	33
pH	8.6	
Na^+ (ppm)	580	10

Pond water contains more salts (chloride, sulfate, bicarbonate, and sodium) than city water and less calcium and magnesium.

Processibility of an oil sand containing 10.75% bitumen has been reported [7] in both deionized water and pond water. A maximum primary recovery of 91% at 0.05 wt% sodium hydroxide was obtained using deionized water, and 95% primary recovery at 0.01 wt% sodium hydroxide was obtained in pond water. The authors concluded that it was significant that the amount of sodium hydroxide needed to obtain maximum primary recovery is less in pond water than in deionized water. This finding explains why sodium hydroxide requirements in industrial use of the process are, in general, much less than sodium hydroxide requirements in the laboratory and in the pilot plant.

The reduced requirement for sodium hydroxide in pond water has been found [8] to be due to the bicarbonate in the water. An average grade oil sand (10.5% bitumen) was studied and was found to give 80% primary recovery at 0.04 wt% sodium hydroxide in deionized water. In pond water, the oil sand gave 93% primary recovery at 0.01 wt% sodium hydroxide. The idea that the bicarbonate in pond water is basic enough to act as a process aid replacement for sodium hydroxide was investigated by processing the oil sand with deionized water containing 13.0 meq/L of sodium bicarbonate. This amount of bicarbonate is close to the amount found in pond water. The processibility curve obtained was very close to the pond water curve with an estimated primary recovery of about 88% occurring at about 0.01 wt%. Surfactant analytical data is included in the report and the authors conclude that bicarbonate is basic enough to act as a process aid in the Clark process and that it acts by producing natural surfactants from precursors in the oil sand.

This effect of water type on the settling rate of tailings from the Clark process has also been investigated [9]. An oil sand containing 9.7% bitumen was processed at optimum sodium hydroxide in both deionized water and pond water and the settling behavior of the tailings were followed with time. The tailings from the experiment using pond water settled much faster (to the 50% point in about 50 days) than the tailings from the experiment using deionized water which required about 200 days before an interface developed. In fact, these tailings had only reached the 90% point at 250 days.

Another study [10] reports sediment volumes after one year for tailings from an oil sand processed at the pilot plant scale using Edmonton city water. Salts (sodium chloride, sodium sulfate, and sodium bicarbonate) were added to a sample of the tailings to bring the concentrations of chloride, sulfate, bicarbonate, and sodium to near those found in pond water. The untreated tailings had settled to the 50% point in one year, while the sample with added salts had settled to the 30% point.

The two studies suggest that tailings produced by the Clark process in pond water, for a given ore, will settle faster than tailings produced in deionized water or river water. Ionic strength is important in determining settling rate.

All the studies available on the effect of water type on processibility and tailings behavior for the Clark process, indicate that data obtained using deionized water or city water are not representative of industrial practice. It is recommended that future laboratory and pilot plant experiments be conducted using pond water.

2.4 Effect of Extraction Parameters on the Process

In commercial practice, bitumen is recovered from oil sand using the Clark process. Oil sand, hot pond water (slurry water), sodium hydroxide, and steam are fed to a rotating tumbler. The slurry produced in this way, is tumbled for about

4 minutes at about 80°C. Once the conditioned slurry exits the tumbler, hot pond water (flood water) is added, rocks and lumps are removed by screening, and the diluted slurry goes to settling vessels where bitumen floats as primary froth and sand sinks as primary tailings. The middle portion of the settling vessel (middlings) contains suspended fines and some bitumen. Both the middlings and the sand from the vessel are further treated, to recover more bitumen, and discharged as whole tailings to settling basins.

The amount of sodium hydroxide used, the temperature of the conditioning stage, the time the slurry is conditioned and the mechanical energy input (tumbler rpm) to the slurry, and the amount of slurry water used are all controllable quantities. The effects of these variables on recovery and on such tailings properties as settling rate, toxicity, and final settled volume are of interest, and several studies have been carried out.

2.4.1 Sodium Hydroxide

Primary recovery for any given oil sand is a function of the amount sodium hydroxide used [1]. Primary recoveries usually start low, increase to a maximum, and then fall off to lower values as the amount of sodium hydroxide used is increased. The amount of sodium hydroxide needed to maximize primary recovery is ore dependent, and a straight line relationship has been shown [1] to exist between sodium hydroxide needed and the fines (44 μm) content of the oil sands. It is possible to predict the amount of sodium hydroxide needed if the fines content of the oil sand is known.

The Fine Tails Consortium has studied [2] the effect of NaOH on tailings sediment volume. The study was a laboratory study, which processed 10 representative oil sands by the published laboratory version [1] of the Clark process, using pond water as both slurry water and flood water. The oil sands were each processed with NaOH (0.02 wt%) and without NaOH. This amount of NaOH in the pond water medium used gave near optimum primary recoveries for all the oil sands. Primary recoveries in the no NaOH case were reduced from the NaOH case, but only by amounts of from 5 to 10%. The tailings from the experiments were placed in graduated cylinders, covered with polyfilm to prevent evaporation, and allowed to settle for one year. The volume of sediment was then determined and expressed as mL of sediment/kg of oil sand. Figure 2-1 shows a plot of volumes for the case using NaOH *versus* the case using no NaOH. The dotted line in the figure represents the expected result if NaOH has no effect on final volume. The experimental data are all close to this line and it is concluded that NaOH has no effect on tailings final sediment volume.

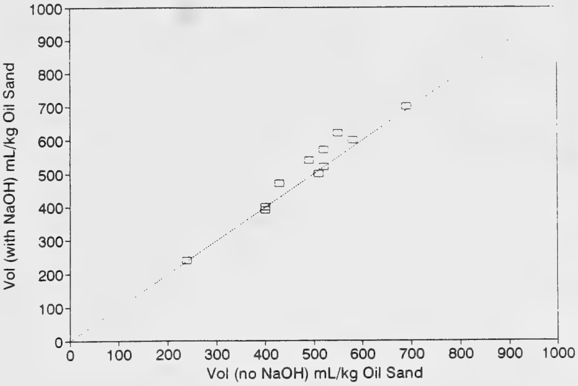

FIGURE 2-1. Effect of NaOH on sediment volume.

Data are also available [11] which permits comparison of tailings sediment volumes for nine representative oil sands processed by the Clark process in pond water using NaOH, and by the OSLO Hot Water Extraction (OHWE) process in river water with Methyl Isobutyl Carbinol (MIBC) and kerosene (no NaOH). The data are shown in Figure 2-2 where the dotted line is the expected line if there is no difference between the two cases. The experimental data are all close to the line and the result confirms that final sediment volume does not differ between a process that uses NaOH and a process that uses no NaOH.

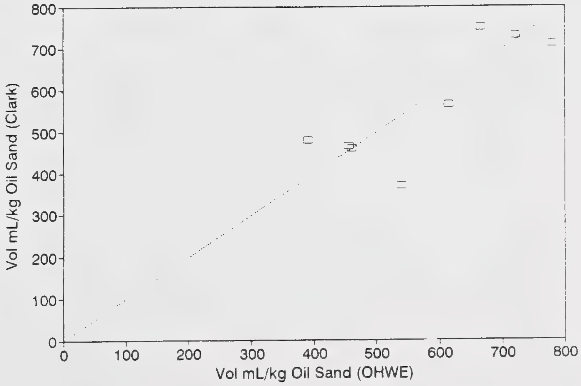

FIGURE 2-2. Effect of process on sediment volume.

Although the data cited above establishes that NaOH does not play a role in determining final sediment volume, NaOH is important when the rate of settling is considered. In one case [12], tailings from an ore processed by the Clark process in pond water without NaOH, required about 10 days to reach 50% settled. Tailings from the same ore processed in the same way but using 0.02 wt% NaOH required about 50 days to reach the 50% point. The final sediment volume, however, was the same in both cases. The NaOH case needed more time to reach the final equilibrium point than the no NaOH case.

The effect of NaOH on tailings toxicity was also studied by the Fine Tails Consortium. In this study, several oil sands were slurried at 80°C for 20 minutes with deionized water, deionized water with 0.03 wt% NaOH, deionized water with 0.06 wt% NaOH, and river water (which contains about 3 meq/L of sodium bicarbonate). The slurries were centrifuged to remove the bulk of the solids and the aqueous phases were ultrafiltered to remove the remaining clay. Toxicities (IC_{50}) were determined by a literature procedure [13]. The data are recorded in Table 2-2.

Several important observations can be made from the above data. Almost all the ores included in the study gave toxic aqueous extracts even when extracted with deionized water. Clearly there is a water soluble, toxic material in many oil sands. Sodium hydroxide made the situation worse, with the extracts using NaOH being more toxic than the extracts using deionized water. This can only mean that NaOH is acting to generate a water soluble toxic material. The use of river water gave extracts which were more toxic than the deionized water extract, but less toxic than the NaOH extracts. It is possible that the bicarbonate in river water is acting to generate toxic materials from the oil sands.

2.4.2 Slurry Temperature

The commercial process is operated at about 80°C and the slurry is held in the tumblers for about 4 minutes. There is some incentive to reduce the operating temperature and the possibility has been investigated using laboratory procedures [14] on an average grade oil sand. The oil sand was first processed by the standard Clark process laboratory procedure at 80°C using the standard slurry time of 10 minutes to establish the optimum NaOH level for the oil sand. Slurry time was then varied from 1 minute to 16 minutes at optimum NaOH. Primary recovery increased with slurry time to a maximum value at 4 minutes, held at this value to 12 minutes and then decreased. At 55°C, primary recovery increased with time to a maximum value at 10 minutes and then decreased.

The results indicate that the Clark process at 80°C requires about 4 minutes of slurry time but that at 55°C, about 10 minutes of slurry time are required.

A study has also been reported of the effect of slurry temperature on fine tails behavior. Settling rate and final sediment volume were identical for an oil sand processed in the laboratory by the Clark process at 80°C and 4 minutes and at 55°C and 10 minutes.

2.4.3 Mechanical Energy

The effect of mechanical energy (as controlled by stirrer speed) on primary recovery from the Clark process at several scales of operation has been reported [15]. The author found that there was a minimum amount of mechanical energy required to produce optimum recovery.

2.5 The Effect of Surfactants on the Process

2.5.1 Theory

A theory which attributes the effect of extraction factors such as ore, water, and process parameters on recovery and tailings behavior to the effect of natural surfactants can be pieced together from evidence in the literature.

TABLE 2-2.

Oil sand	IC_{50} DI water	IC_{50} 0.03 NaOH	IC_{50} 0.06 NaOH	IC_{50} River water
8804	39	20	14	29
8801	42	18	14	32
8807	39	13	10	28
8806	100	16	18	100
8812	36	15	12	39
8901	81	19	12	38
8907	100	30	19	59
8910	72	34	20	70

According to this theory, natural surfactants are produced when oil sands are processed by the Clark process using NaOH [16]. The surfactants are the agents that act to optimize primary recovery from the process [17]. They do this by lowering interfacial tensions and increasing interfacial charges for all the phases involved in the process (oil, water, and solids). The increased charges on both the bitumen droplets and the solid particles favor bitumen detachment from the solids, while lowered interfacial tension of the bitumen droplets and the air bubbles favors attachment of bitumen to air. In fact, the prediction was made [17] that bitumen will engulf the air bubbles because the much decreased surface area of the resultant bitumen coated air bubble is thermodynamically favored.

There is a critical concentration of natural surfactants [18], the same for all oil sands, which must be achieved to optimize primary recovery. This concentration is about 0.12 meq/L as measured in tailings from the process or about 60 ppm, assuming a molecular weight of 300 for the surfactants. The surfactants found in tailings water are believed to be in equilibrium with surfactants adsorbed at the various interfaces and the critical concentration is the concentration needed in the water to get maximum charges at interfaces. The charge on bitumen particles has been shown [19] to go through a maximum at the critical concentration, and the charge on clay has been shown to reach a maximum value at the critical concentration.

The natural surfactants are a mixture of naphthenates [20] and sulfonates [21]. Information is available about the chemical structure of naphthenates [22], but nothing is available about the chemical structures of sulfonates.

It has been hypothesized that natural surfactants are responsible for the slow settling behavior, toxicity, and the difficulty of compaction of Clark process tailings [11]. It is suggested that some of the natural surfactants produced by the sodium hydroxide in the extraction step of the Clark process, end up adsorbed on the clay particles in the fine tailings. The clay particles, as a result, have a high negative charge and are bulky (or fuzzy). A combination of electrostatic and steric repulsions inhibits settling and accounts for the slow settling behavior of the tailings. The same forces are believed to make these tailings difficult to compact by mechanical methods. In addition, it has been shown [23], that one of the surfactants (naphthenate) is toxic. It is this surfactant that produces the toxicity of the tailings.

The assumption is usually made that if a process could be devised which did not require sodium hydroxide, surfactants would not be produced. However, this assumption is not correct. Surfactant production as a function of sodium hydroxide added, has been determined for several ores and these studies have included blank extractions using no sodium hydroxide. Many of the ores (see for instance [18]), gave tailings containing appreciable concentrations of natural surfactants when extracted by deionized water. However, some ores give tailings free of surfactants under these conditions. The finding, previously discussed in the section on the effect of NaOH on the process, that many oil sands give deionized water extracts that are toxic, confirms that ores do exist that produce water extracts containing natural surfactants even when NaOH is not used.

Both naphthenates and sulfonates are known to react with calcium compounds to form insoluble (and hence inactive) calcium salts. If it is true that the natural surfactants are responsible for the slow settling, toxicity, and difficulty in compaction of Clark fine tailings, then the possibility exists that the addition of small amounts of calcium compounds will make the tailings fast settling, non toxic, and easy to compact.

The theory has been used for instance [24] to explain the relationship discussed earlier between the fines content of oil sands and the amount of NaOH needed to get maximum primary recovery (the more fines the more NaOH needed). According to this explanation the fines fraction of oil sands contains compounds such as magnesium sulfate, calcium sulfate, magnesium carbonate, and calcium and magnesium clays. The greater the amount of fines, the greater the amount of magnesium and calcium compounds. The calcium and magnesium compounds act to consume NaOH in competition with the reaction(s) that produce natural surfactants. Consequently, the greater the fines content, the greater the amount of NaOH required to produce the critical concentration of surfactants.

Process parameters such as temperature, time, amount of NaOH, and amount of slurry water are, by the theory, seen to be interrelated because the purpose of the conditioning step of the process is to produce the critical concentration of natural surfactants. All of these parameters would be expected to influence the rate of the reaction that produces surfactants.

The observation that a decrease in slurry temperature requires an increase in slurry time, is explained as a kinetic effect. The rates of the reactions producing surfactants, are reduced by the temperature decrease, and extra time must be allowed to produce the critical concentration. The requirement for a minimum amount of mechanical energy occurs because the surfactant producing reactions are heterogenous. Reaction between NaOH in the aqueous phase and precursors in either the bitumen or the solids requires stirring to bring the reactants into contact with each other.

There is not much experimental evidence in the literature to confirm that natural surfactants act to maximize recovery and are responsible for the poor settling behavior of tailings. Some work was done by the Fine Tails Consortium in an attempt to confirm that surfactants are present in tailings water from the Clark process, that they have some influence on primary recovery and that they have an effect on tailings properties. The research began with development of a method to isolate natural surfactants from the Clark process tailings water and proceeded to study the effect of the surfactants on recovery and on fine tailings properties.

2.5.2 Isolation of Surfactant from Pond Water

Many attempts have been made over the years to isolate and identify the surfactants in Clark fine tailings. In the first reported attempt [17], a surfactant fraction was isolated from tailings by foam fractionation and the surfactant was identified as a carboxylate by infrared analysis. There was a carboaylate peak at 6.4 μm which shifted to a carboxylic peak at 5.8 μm on acidification. Somewhat later the carboxylate from foam fractionation was identified [20] as a naphthenate.

In another study [21], the surface active fraction was isolated from tailings water by foam fractionation followed by azeotropic removal of the water from the concentrate with toluene. The toluene was then concentrated to a small volume and a toluene insoluble fraction (the surfactant salts) was collected by filtration. The salts were treated with hydrochloric acid and water and the resulting solution was extracted with ether. The ether extract contained a carboxylic acid according to the infrared spectrum. The salts were also shown to contain a sulfonate by treatment with phosphorous pentachloride which gave a product with characteristic sulfonyl chloride bands in the infrared. Treatment of this product with ammonia gave another product with characteristic sulfonamide adsorption bands in the infrared.

A procedure for the isolation of surfactants from pond water was developed based on the literature. If naphthenates and sulfonates are the only species present, then it should be possible to isolate the naphthenate (as naphthenic acid), by adjustment of pond water pH to approximately 3 and extraction with methylene chloride. This procedure would be expected to succeed because sulphonates do not convert to sulfonic acids at pH 3 (they are very weak bases and need a pH of about 1.0 to convert to the acid form). Consequently, the naphthenates would be expected to be in the acid form, and hence soluble in methylene chloride at pH 3, while the sulfonates would be expected to be in the salt form, and hence insoluble in methylene chloride at pH 3.

Carboxylic acids were initially isolated [25] from pond water by acidification of ultrafiltered pond water to approximately pH 3 and extraction with methylene chloride. The pond water was ultrafiltered using a ultrafilter designed to hold back materials of MW greater than 5000. This ultrafilter was chosen to remove bitumen and clay from pond water while allowing surfactants and salts to pass through. It became apparent, after some experience with this procedure, that the carboxylic acids were too soluble in water to be completely extracted from water by methylene chloride. Accordingly, the procedure described in the Appendix was adopted. In this procedure the aqueous phase, after acidification, is concentrated to dryness and the dry residue is extracted with methylene chloride. After concentration of the extract to dryness, a brown oily product was obtained which was identified by FTIR and FAB MS as naphthenic acid. Naphthenic acid has been shown [22] to consist of a mixture of saturated straight chain and cyclic carboxylic acids. The FAB MS of pond naphthenic acid had the same parent peaks as the FAB MS of Eastman naphthenic acid, only moved to slightly lower masses.

Extraction of dried, acidified pond water with methylene chloride did not recover all the organic material and an effort was made to develop a procedure for the isolation of the remainder of the organics. Several solvents were investigated at several temperatures and it was found that what appears, from the FTIR spectrum, to be an organic hydroxy carboxylic acid could be recovered by extraction with room temperature anhydrous ethanol.

A third organic fraction was obtained from the residue after the cold anhydrous ethanol extraction by extraction with boiling anhydrous ethanol. This fraction appeared to be a sodium sulfonate from the FTIR spectrum.

An organic material mass balance was done on the experiment. The ultrafiltered pond water had TOC of 120 mg/L, and if we assume the organic compounds in the pond water are roughly 80% carbon, then the amount of organic material in pond water is 150 mg/L. The experiment gave 125 mg/L of naphthenic acid, 42 mg/L of hydroxy carboxylic acid, and 61 mg/L of sulfonate. The total of isolated organics was 228 mg/L, which is considerably more than the estimated 150 mg/L based on TOC of the feed pond water. Sodium chloride and sodium sulfate should be present in the dried residue and both of these are known to have some solubility in ethanol. They are both soluble to a slight extent in cold ethanol and more so in hot ethanol. The excess weight of isolate is probably due to contamination of the hydroxy carboxylic acid and the sodium sulfonate with these two salts. The residual solids were dissolved in water and TOC was determined in the water. There was a very small amount of organics in the water amounting to about 1.2 mg/L of feed pond water. The procedure appears to recover all the organics, but it is probable, that the fractions obtained by ethanol extraction are contaminated with sodium chloride and sodium sulfate. Naphthenate appears to be the major organic present in pond water. The water recovered during concentration of the acidified pond water had TOC of 5.8 mg/L for a carbon loss of about 15 mg. This loss is thought to be naphtha, which is known to be present in the pond.

The theory that surfactants act to optimize recovery from the hot water process and control the properties of fine tailings calls for the surfactant to be present in the sodium salt form. A procedure for the conversion of naphthenic acid to sodium naphthenate was devised and is given in the Appendix.

2.5.3 Effect on Primary Recovery

It was considered that the theory would be proven if it could be shown that the addition of natural surfactants to the slurry stage of the Clark process caused an increase in primary recovery similar to that produced by sodium hydroxide. Since the theory suggests that natural surfactants act to optimize recovery at very low concentrations, the experimental program was designed to test low concentrations.

Commercial sodium naphthenate, pond sodium naphthenate, and the total mixture of pond surfactants were tested for their effect on primary recovery on the Clark process in deionized water [26]. The additives were tested at concentrations near, and slightly above, the critical concentration. There was no improvement in primary recovery in any of the experiments. However, there is evidence [1] that surfactants can produce increases in primary recovery if added to the slurry at higher concentrations. Tide, sodium oleate, and lignosulfonate have been tested [1] and all were found to increase primary recovery at the 0.1 wt% on the oil sand level. This quantity of sodium oleate would be expected to produce a tailings concentration of about 1.5 meq/L or about 10 times the critical concentration. One possible explanation for the failure of natural surfactants to act at low concentrations, may be that large quantities are adsorbed at interfaces. A second explanation, may be that the surfactants are consumed by calcium compounds, particularly calcium clays, which may occur in oil sands [24]. Calcium clays, if present, would be expected to react with sodium carboxylates to yield sodium clays and calcium carboxylates, both of which would be expected to be insoluble. The proposed reaction is shown in Eq. (3).

$$(3)\quad \mathrm{Ca(clay)_2 + 2RCOONa \rightarrow 2Naclay\downarrow + RCOOCaOOCR\downarrow}$$

If the second explanation is correct, then it should be possible to use low concentration of natural surfactants if the process is operated in pond water. The use of pond water should solve the problem because the carbonate/bicarbonate mixture in pond water would be expected to react with calcium clays according to the reaction shown in Eq. (4).

$$(4)\quad \mathrm{Ca(clay)_2 + Na_2CO_3 \rightarrow 2Naclay\downarrow + CaCO_3}$$

Consumption of calcium clays by carbonate/bicarbonate would be expected to reduce consumption of sodium carboxylates by calcium clays and make it possible to operate the process at low surfactant concentrations. A series of batch extractions were conducted using pond water as both slurry water and flood water. A relatively low grade oil sand (7.8 wt% bitumen) was selected for the experiments as it is the low grade oil sands that show the biggest response to sodium hydroxide when pond water is used. The oil sand was processed in pond water with no additives, with the optimum amount of sodium hydroxide and with various quantities of Kodak sodium naphthenate. The data are available [26].

The pond water used in the experiments was analyzed for bicarbonate and was found to contain 16.0 meq/L. The tailings water from the experiment in which no additives were used was also analyzed for bicarbonate and was found to contain 13.0 meq/L of bicarbonate. This result indicates that the oil sand involved has some ability to consume bicarbonate and the result is in agreement with the hypothesis that calcium compounds in oil sands can be neutralized by the bicarbonate in pond water if the process is carried out in pond water.

The oil sand gave about 40% primary recovery when processed without any additives and 61% primary recovery with 0.02 wt% sodium hydroxide. Kodak sodium naphthenate was tested at concentrations of 0.12, 0.24, 0.36, and 0.48 meq/L. The first three concentrations gave primary recoveries of about 50% while the 0.48 concentration gave a primary recovery of 45%. The 50% primary recovery was up by 10% from the no additive result but was short of the 60% obtained using 0.02 wt% sodium hydroxide.

The result with Kodak sodium naphthenate was encouraging because it indicates that the hypothesis that calcium compounds can be prevented from precipitating naphthenates by using pond water is correct and because the finding confirms that the hot water process can be operated at very low surfactant concentrations. It appears that there is more to the story than naphthenates alone because the 60% primary recovery obtainable with sodium hydroxide could not be obtained with sodium naphthenate.

Further experiments were carried out with another low grade oil sand, which had a bitumen content of 8.14 wt%. This oil sand was processed in pond water with no additives, with sodium hydroxide and with various concentrations and combinations of the surfactants isolated from pond water. The no additive experiment gave a primary recovery of 15%. Sodium hydroxide gave a maximum primary recovery of 28% at 0.02 wt%.

The bicarbonate input (in the pond water) to the no additive experiment, was determined to be 14.83 meq and the bicarbonate output (in the total tailings water), was determined to be 12.69 meq. There was, as expected, some consumption of bicarbonate in the experiment. The amount consumed was 2.14 meq and it was determined that the amount of bicarbonate at the slurry stage of the process was only 2.11 meq. This result indicates that there was insufficient bicarbonate present at the slurry stage to consume all the calcium compounds in this oil sand and that consequently, consumption of added carboxylate surfactants could be expected to occur and invalidate the experiments. A decision was made to proceed with this oil sand, adding extra bicarbonate at the slurry stage as required. Consequently, some experiments were carried out with added bicarbonate to determine how much would be required to neutralize the calcium compounds. The experiments showed that about 200 mg of sodium bicarbonate was necessary. Primary recovery, using this amount of bicarbonate, was, at 17%, almost unchanged from the blank experiment.

A series of experiments were then carried out in pond water with added sodium bicarbonate and various combinations and amounts of surfactants isolated from pond water. Pond naphthenate alone gave primary recoveries of about 23 to 24%, up from the 17% obtained with bicarbonate alone. There was little difference in primary recovery between 0.17 meq/L and 0.26 meq/L. The increase in primary recovery is judged to be experimentally significant and, again, is an improvement, but it did not equal the primary recovery obtainable with sodium hydroxide (28%).

The pond hydroxy carboxylic acid fraction was also tested and found to improve primary recovery slightly over the sodium bicarbonate case, but the recovery improvement was not as great as that obtained with sodium naphthenate. Two concentrations of the material were tested, 0.14 and 0.24 meq/L. Both gave about the same primary recovery at 19 to 20%, which is up from 17% but not as good as the 23 to 24% obtainable with sodium bicarbonate.

The pond sulfonate fraction was tested at four concentrations ranging from 0.21 to 1.60 meq/L. Primary recovery showed a small peak at 0.38 meq/L where the primary recovery was about 21%. Excess pond sulfonate was detrimental, causing primary recovery to decrease to about 13%.

An experiment was carried out using all three surfactants in proportion to the actual quantities isolated from pond water. Using 0.23 meq/L of pond naphthenate, 0.12 meq/L of pond hydroxy carboxylate, and 0.28 meq/L of pond sulfonate resulted in a primary recovery of 33%. This result represents success, as it is better than the 28% obtainable with sodium hydroxide. The concentration of sodium naphthenate used in the experiment at 0.23 meq/L is more than the 0.12 meq/L predicted [17] and probably means that the extra quantity is the quantity needed to saturate interfaces in the system. The result also means that there is a synergistic effect, with the mixture of surfactants performing better than any of the individual components.

It is concluded that surfactants are needed at concentrations near the amount predicted in the literature [18] to maximize primary recovery from the Clark process.

2.5.4 Effect of Surfactants on Tailings Behavior

Theory [11] suggests that natural surfactants, adsorbed on clay particles, are responsible for slow settling, toxicity, and difficult compaction of fine tailings. Tailings with these adverse properties are almost always produced by the Clark process and are sometimes (depending on the ore) produced by the OHWE process. The experimental data, on which this theory is based, are discussed in this section. Settling rate data (defined as the time needed for a tailings sample to settle to the 50% point) are available [11] for several representative Syncrude and Suncor ores processed by the OHWE process. There was a large variation in settling rates with some samples settling in less than one day, while others required as much as 200 days. The theory suggests that the variation is due to variation in surfactant concentrations, and a plot of settling rates *versus* surfactant concentrations would be expected to yield a straight line with high surfactant concentrations related to slow settling. However, at the time the work was carried out, there was no easy, reliable way to measure surfactant concentrations in tailings. However, electrophoretic mobilities (EM) of the fine particles in the tailings were measured and are known [19] to be directly related to surfactant concentrations. Consequently, a plot of settling rates *versus* electrophoretic mobility would also be expected to yield a straight line. Since electrophoretic mobility is also known to depend on ionic strength, the success of this treatment of the data involves a requirement that ionic strength be almost constant through the series. This requirement is met for the data from the OHWE experiments which were all carried out in river water. Figure 2-3 shows a plot of settling time *versus* electrophoretic mobility for all the OHWE experiments.

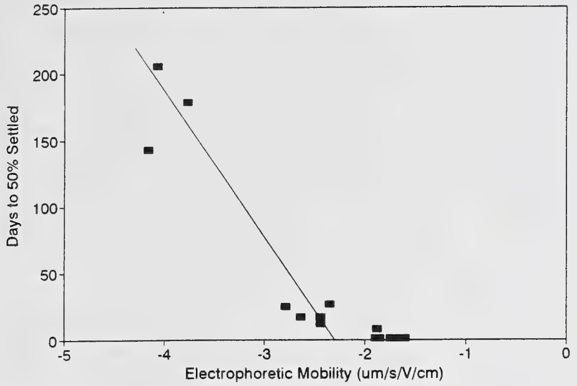

FIGURE 2-3. Effect of EM on OHWE settling rate.

A fairly good straight line relationship between settling time and electrophoretic mobility was found for mobilities more negative than –2.3 μm/s/V/cm. Samples with electrophoretic mobilities less negative than –2.3 were all fast settling (less than one day). It is suggested the tailings samples with electrophoretic mobilities less negative than –2.3 were free of surfactants. The linear relationship between settling rate and electrophoretic mobility agrees well with the proposal that natural surfactants are responsible for slow settling.

Electrophoretic mobility is plotted *versus* calcium concentration for the data from the OHWE experiments in Figure 2-4. Samples which settled fast (less than one day) are shown as open squares, while samples that were slow settling (more than one day) are shown as solid triangles. The figure indicates that electrophoretic mobility decreases to a constant minimum of about –2.0 once calcium concentration reaches about 7 ppm. At this level, all the surfactants are gone, and further amounts of calcium cannot further reduce the electrophoretic mobility. Figure 2-4 also shows that tailings with calcium concentrations above about 7 ppm, and electrophoretic mobilities at the minimum are the ores that are fast settling. The fast settling behavior is explained as due to the absence of natural surfactants.

If surfactants are really responsible for slow settling, then the addition of calcium compounds to slow settling tailings should reduce natural surfactant concentration and make electrophoretic mobility less negative and the rate of settling fast. The effect of calcium chloride on electrophoretic mobility was examined [27], the data being shown in Figure 2-5. Electrophoretic mobility of fine particles in the tailings sample studied, decreased from –2.8 to –0.9 μm/s/V/cm in agreement with the theory. The effect of calcium sulfate on settling rate was examined [28] for several slow settling tailings samples at a calcium sulfate addition level of 125 ppm. The data are tabulated in Table 2-3.

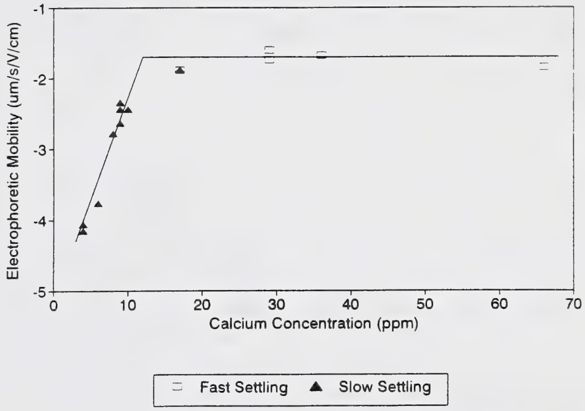

FIGURE 2-4. Effect of EM, Ca on settling (OHWE).

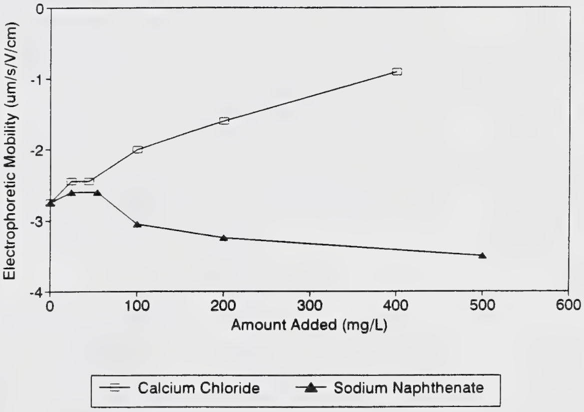

FIGURE 2-5. Effect of additives on EM.

Settling rates were greatly enhanced by the calcium sulfate as the theory predicts. Calcium sulfate treatment did not have any effect on the final sediment volumes which, after one year, were the same for the treated samples as for the untreated samples.

The addition of surfactants to fast or moderate settling tailings would be expected to make the electrophoretic mobility more negative and slow the settling rate. The effect on electrophoretic mobility was examined [27] by the addition of sodium naphthenate to the same tailings used in the study with calcium chloride. The results are also shown in Figure 2-5. Sodium naphthenate made the electrophoretic mobility more negative (from –2.8 to –3.5 μm/s/V/cm) in agreement with the theory. The effect of surfactants on settling rate was examined by adding sodium naphthenate and sodium dodecane sulfonate to tailings from an ore known to give fast settling tailings. The data are tabulated in Table 2-4. The surfactants did act to slow the settling rate of the tailings in confirmation of the theory.

The theory also suggests that natural surfactants are responsible for the toxicity of fine tailings from the Clark process, and if correct, a relationship should exist between natural surfactant concentration and toxicity. There should also be a relationship between electrophoretic mobility of the fine particles in tailings and toxicity, provided ionic strength remains reasonably constant. The data from the OHWE experiments are shown in Figure 2-6. A straight line relationship was found between electrophoretic mobility and toxicity for electrophoretic mobilities more negative than –2.0 μm/s/V/cm. Samples with electrophoretic mobilities less negative than –2.0 were nontoxic and it is believed that this is because these samples are surfactant free.

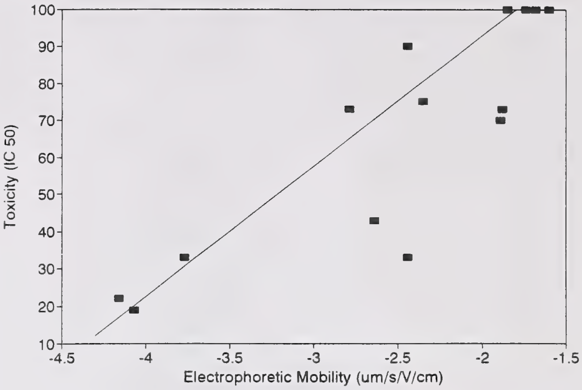

FIGURE 2-6. Effect of EM on IC_{50} for OHWE tails.

A plot of electrophoretic mobility *versus* calcium concentration for the OHWE experiments is shown in Figure 2-7. Samples which were relatively nontoxic (IC_{50} of 100) are shown as open squares while samples of $IC_{50} < 100$ are shown as solid triangles. Samples with calcium concentration greater than 7 ppm and electrophoretic mobility at the minimum of about –1.7 μm/s/V/cm were nontoxic. It is thought that these samples are surfactant free.

TABLE 2-3.

Oil sand	Settling rate (days) untreated	Settling rate (days) treated
8804	27	21
8806	146	14
8807	17	2
8812	25	4
8901	153	7
8907	163	30

TABLE 2-4.

Sodium naphthenate added (meq/L)	Sodium dodecane sulfonate added (meq/L)	Days to form interface
0	0	13
0.40	0.18	21
0.40	0.37	27
0.80	0.37	55+
0.80	0.74	55

TABLE 2-5.

Oil sand	Toxicity IC_{50} untreated	Toxicity IC_{50} treated
8804	75	100
8806	22	100
8807	33	100
8812	73	100
8901	19	100
8907	33	100

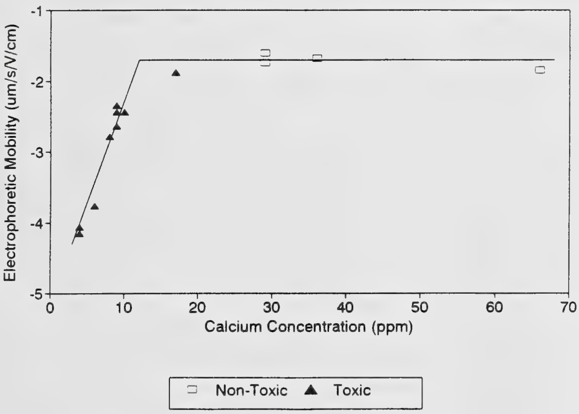

FIGURE 2-7. Effect of EM and Ca on IC_{50} (OHWE).

The addition of calcium compounds to tailings which contain natural surfactants (and hence are toxic), would be expected to remove the surfactants and make the tailings nontoxic. Experimentally, samples of tailings (toxic) produced by the OHWE procedure were treated with calcium sulfate (125 ppm), with representative results in Table 2-5. In all cases, treatment with calcium sulfate made the samples relatively nontoxic.

The data relating surfactant concentrations to toxicity, as discussed above, confirm that natural surfactants are responsible for the tailings toxicity.

The theory also suggests that compaction of Clark fine tailings is difficult due to electrostatic and steric effects. If the theory is correct, treatment with calcium compounds would be expected to remove the surfactants from the fine particles and make the tailings easy to compact.

A test was designed to measure ease of compaction of fine tailings by measuring centrifugal force needed to centrifuge the fine solids in the sample down to a cake. Samples were centrifuged first at 1000 rpm for 20 minutes and the supernatant poured off, the light transmittance of the supernatant measured, and the weight of residue determined. The sample was then recombined and the procedure repeated at 2000 rpm, increasing in increments of 2000 rpm up to 10000 rpm.

The test procedure was used on two samples, one untreated tails from the Clark process, and the other the same tails after treatment with calcium sulfate. The data are shown in Table 2-6. The untreated tails needed about 8000 rpm to centrifuge down the solids. The weight of cake was maximum at 8000 rpm and light was first transmitted through the sample at 10000 rpm. The solids in the treated tails were centrifuged down easily with all the solids down at 1000 rpm and light transmittance first occurring at 2000 rpm.

The result lends support to the theory and suggests that treatment of Clark tailings with calcium sulfate may lead to an economically viable way to compact these tailings for disposal.

2.6 Conclusions

1. Both processibility and the behavior of tailings from the Clark process are ore dependent. The amount of NaOH needed to obtain maximum primary recovery is dependent on the fines content of the oil sand (<44 μm), while the final sediment volume is dependent on the clay content of the oil sand. Clay content is best determined by measuring either methylene blue uptake or exchangeable potassium.
2. Both processibility and the behavior of tailings from the Clark process are dependent on the type of water used in the process. The use of recycle pond water rather than deionized water or city water permits a reduction in the amount of NaOH needed for maximum primary recovery and leads to tailings which settle faster.

TABLE 2-6.

Centrifuge speed (rpm)	Untreated % transmission	Treated % transmission	Untreated wt. sediment	Treated wt. sediment
1000	0.5	0.6	1.7	33.9
2000	0.5	16.1	4.2	22.7
4000	0.5	65.9	5.3	14.7
6000	0.5	71.3	7.2	12.1
8000	0.5	89.0	8.4	9.8
10000	26.9	88.1	8.1	9.3

3. Processing conditions such as NaOH quantity, slurry temperature, slurry time, and mechanical energy input have important effects on processibility and tailings behavior from the Clark process. The inputs of NaOH, temperature, time, and mechanical energy must be balanced to optimize recovery and these inputs are seen as interrelated. The use of sodium hydroxide leads to tailings which settle slower and are more toxic than tailings from processes that do not involve the use of NaOH. However, the use of NaOH does not result in a greater volume of final sediment.
4. Natural surfactants are produced at the conditioning phase of the Clark process and they act to maximize primary recovery and are responsible for some of the undesirable properties of fine tailings from the process. Settling rates become slower, toxicity increases, and the fines become more difficult to compact as the natural surfactant content of the tailings increases.
5. Natural surfactants can be removed from tailings by the addition of small amounts of calcium sulfate. This treatment causes slow settling, toxic, and difficult to compact tailings to become fast settling, nontoxic, and easy to compact.

2.7 Recommendations for Future Work

The results described in this chapter indicate that treatment of slow settling, difficult to compact tailings with calcium sulfate can make the tailings fast settling and easy to compact. It may be that an economically viable commercial method for tailings disposal can be developed based on this discovery. Research at both the laboratory scale and pilot scale will be needed to further develop the method.

It will be important, if the method is to be commercialized, that the aqueous phase from the calcium sulfate treatment can be reused in the extraction process. This question can be addressed at the laboratory scale. The amount of calcium sulfate used will need to be controlled so that the desired results are achieved while holding the bicarbonate content of the tailings at some steady state and this quantity needs to be determined. Too little calcium sulfate will not achieve the desired results while too much would be expected to remove all the bicarbonate from the water and allow the calcium concentration to build up. A balance needs to be struck between bicarbonate removal by the calcium sulfate and bicarbonate renewal by using the treated water and NaOH to process more oil sand.

It will also be important, if the method is to be commercialized, to develop an economically viable way to compact the tailings after calcium sulfate treatment. The laboratory work indicates that, although the solids are easier to compact, the compaction cannot be achieved by the force of gravity alone. Centrifugation will probably be too expensive but the sand compaction process developed by the OSLO Consortium may work and may be economically viable. Engineering research at the pilot and field scale will be required.

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2.9 Appendix

2.9.1 Conversion of Pond Naphthenic Acid to Sodium Naphthenate Solution

Pond carboxylic acid (259 mg) was transferred to a beaker (500 mL) and deionized water (200 mL) was added. The beaker was fitted with a magnetic stirrer bar, a pH probe, a nitrogen dispersion tube, and a thermometer. It was placed on a hot plate and brought to about 45°C with stirring under nitrogen. Sodium hydroxide solution (0.1 N) was added dropwise to bring the pH from about 3.5 to about 9.0. Stirring was continued until the pH of the solution dropped to about 8.0. The pH was again adjusted to 9.0 and the process of waiting for the pH to fall and readjustment was repeated until the pH stabilized at about 8.7. One day was required to dissolve all the brown oily carboxylic acid to form a yellow solution (211.8 g). A sample (25 mL) was diluted to 40 mL with deionized water and titrated with hydrochloric acid (0.0187 N). There was a first end point at pH 8.0 (species concentration 0.066 meq/L), and a second end point at pH 4.3 (species concentration of 4.94 meq/L).

2.9.2 Isolation of Natural Surfactants from Pond Water

Pond water (4 L) was ultrafiltered using a Amicon Model 8200 ultrafiltration cell fitted with a YM3 Diaflow ultrafilter (nominal retention of 3000 MW). A portion of (3023.0 g) the ultrafiltered pond water was weighed to a 4 L beaker and the beaker was fitted with a magnetic stirrer and a pH electrode. The pH of the solution was then adjusted to about 3 by adding 6 N hydrochloric acid in a dropwise manner. The solution turned cloudy during the addition and remained cloudy after the addition was complete. The acidified pond water was concentrated to dryness using a rotary evaporator operated at full house vacuum with the water bath at 60°C using a 1 L round bottom flask. An off white solid residue (5.60 g) was obtained. The round bottom flask was fitted with a magnetic stirrer and a reflux condenser, dichloromethane (250 mL) was added and the apparatus was placed in a heating mantle. The mixture was refluxed gently for 20 minutes. At the end of this time, the liquid was decanted from the solids. Some solids carried over with the clear liquid were collected by filtration and returned to the round bottom flask. The procedure was repeated with another 250 mL portion of dichloromethane and the extracts were combined and concentrated to dryness on a rotatory evaporator. A dark yellow oil (291 mg) was obtained. The material was identified as naphthenic acid. Anhydrous ethanol (100 mL) at room temperature was added to the residue in the 1 L round bottom flask and the mixture was stirred with the magnetic stirrer for 10 minutes. The aqueous phase was recovered by decantation and a small amount of solids, which decanted with the aqueous phase, was recovered by filtration and returned to the round bottom flask. The ethanol extract was concentrated to dryness using a rotary evaporator to yield 130 mg of an orange oil. The material, according to the IR, was a hydroxy carboxylic acid. Anhydrous ethanol (100 mL) was added to the residue in the 1 L round bottom flask and the mixture was refluxed for 20 minutes. At the end of this time the mixture was decanted, while still hot, through filter paper. The solids collected on the filter paper were returned to the 1 L round bottom flask and the extraction was repeated once more with another 100 mL of anhydrous ethanol. The ethanol extracts were combined and concentrated to dryness using a rotatory evaporator to yield 302 mg of a white crystalline material. The material according to the IR was a sulfonate.

3.0 FINE TAILINGS FORMERS

3.1 Introduction

Before the advent of the Fine Tailings Fundamentals Consortium many years of research and engineering had failed to produce an economical process for reducing the volume of oil sands fine tailings (MFT). Then, it was felt that the use of sodium hydroxide in the Clark Hot Water Extraction (CHWE) process was necessary to disperse bitumen into fine droplets to maximize their recovery. It was concluded that the tailings problem was inherent to, and a direct result of, the extraction process used.

The most obvious difference between MFT and other industrial tailings lies in the oil content of MFT. Removal of the oil simultaneously removes iron compounds, the bulk of the treated fine tailings then settle, relatively rapidly, to a denser material. It was thus natural to assign a major role to an "iron-organic complex" as a cause of MFT volume. While these observations did not constitute proof that such a "complex" is responsible for the water holding capacity of MFT the concept made a convincing starting point for research into methods of altering MFT to reduce its volume. Thus, there was a program to enhance the transformation and crystallization of iron hydroxides into denser iron compounds through the addition of complexing ligands, such as succinate. Others attempted to precipitate amorphous iron as the more readily compactable sulfide.

An important insight into the structure of fine tailings is contained in the paper by Yong and Sethi [1]. A major outcome of this work was the concept that the specific volume of fine tailings is the weighted sum of the specific volumes of the mineral constituents of the tailings. Measurements were made for model tailings constituents such as kaolinite, mica, mixed layer clays, amorphous iron oxide, and others. The relative abundance of these constituents in an average Suncor tailings sample was determined. The cumulative volume of tailings was calculated based on these values. This work shifted the perspective on the cause of MFT volume to amorphous materials, particularly iron oxide, and fine clays, such as bentonite (montmorillonite). However, significant quantities of these minerals have never been detected in fine tailings.

By the time the consortium work started it was understood that the fine tailings volume was determined by the make-up of the mineral particles present. The particles were not individually suspended in the water phase but occurred in an aggregated state. Fine clay particles flocculate without much regard for the attainment of a minimum energy configuration. Once the flocs are formed and start to settle they begin to interfere with each other and the progress of volume reduction becomes minimal. The particles may, however, slowly reorganize themselves into lower energy positions. A settling equation for tailings, based on the time dependence of the fractal dimension of these aggregates, was developed at that time [3]. This equation, with only one adjustable parameter, describes laboratory settling experiments with great accuracy. While the basis for understanding tailings behavior was in place, no information was available on the components involved, their origin, how their interaction was affected by water chemistry, and the correlation with tailings volume. These questions provided the impetus for the development of methods to separate fine tailings into its component parts to allow individual study of their properties.

During later work the perceived importance of specific minerals declined when it became clear that ultra-fine clay particles were ubiquitous in oil sands tailings and ores; delaminated kaolinite and mica are the most prevalent minerals. Many of these clay particles are only a few layers thick and single layer clays are not uncommon. The mineralogy of these ultra-fine particles is difficult to determine as they are often too small to induce interference in reflected X-rays. Consequently, ultra-fines appear to be largely amorphous when examined by X-ray diffraction. However, investigations carried out during the consortium mandate have shown beidellite [2] to be the most important structure forming tailings constituent.

Recently, measurements on model colloidal systems have been made to determine the rate of floc formation. Study of the resulting structures have confirmed experimentally that flocs are fractal objects [4] and that fractal dimension is a measure of floc density [5]. These measurements show that the latter decreases with increase in floc radius. Also, it has been demonstrated that the rate of floc formation affects the fractal dimension of the resultant flocs [6,7]. Flocs formed quickly have a very low density compared to those formed slowly. Also, flocculation in a dilute suspension leads to less dense aggregates compared to the same process carried out at a higher concentration of suspended solids. Another consequence of the fractal nature of flocs is that they become denser following breakdown and reattachment of the fragments [8]. This is an elegant explanation for the observation made by many workers that MFT, after stirring, will release water while the broken flocs are in the process of rearrangement.

An improved understanding of the fundamental role of ultra-fines in MFT formation has provided some insight into possible solutions to the tailings problem. One simple operational strategy is to run bitumen extraction processes at as high a solids loading as possible to minimize tailings volume. This is entirely equivalent to processes designed to increase capture of fines in the tailings pond beaches and dykes. A second approach is to prevent tailings formation by suppressing the flocculation and gelation of ultra-fines. By preventing the viscosity increases associated with gelation, coarser clay particles are allowed to settle out with the coarse sand on the pond beaches. The low viscosity of recycle water, containing dispersed ultra-fines, allows it to be reused without adversely affecting bitumen recovery. This is the approach advocated by the proponents of the ZEFTE process [9].

Finally, the current concept of MFT, wherein the material is a gel or three-dimensional entanglement of flocs, leads to an interesting extrapolation as to what would happen if the water soluble, stabilizing ions are removed from the system. This experiment has been done and indeed MFT does lose strength [10]. Coarse particles start to settle through the MFT and the ultra-fines become dispersed. This so-called leaching of MFT is of importance for the design of disposal schemes where the MFT is stored in deep lakes, capped by clean surface run-off water.

3.2 Ultra-Fines Separation and Distribution

3.2.1 Principles

The gel-like structure of CHWE fine tailings is relatively weak and it undergoes shear thinning to produce a more fluid material; on cessation of mixing the gel structure re-forms. The tailings therefore exhibit the property of thixotropy. However, the time scale of this process is such that the system will gel before coarser solids, initially entrapped in the structure, can be released. Therefore, even after vigorous agitation, the tailings structure will return to its original state with almost no change, that is, to a visually non-segregating mixture. Nevertheless, if the agitation step is followed by mild centrifugation then the sedimentation rate for the individual components is increased sufficiently for fractionation to occur. An example of this behavior is shown on Figure 3-1, where the photographs compare a sample of untreated tailings with one previously subjected to vigorous agitation and then settled under mild centrifugation conditions.

The Cold Water Agitation Test (CWAT) was developed as a simple method to allow fine tailings to be fractionated into its component parts. In general, this treatment causes the tailings sample to separate into four distinct layers (again see Figure 3-1); from bottom to top these are:

(a) A light colored sediment containing the coarser solids, originally entrapped in the gel structure (RS).

(b) A dark colored layer (ORS) with significant insoluble organic carbon and heavy metal content.

(c) An aqueous ultra-fine suspension (A), containing two gel forming components (HUS and BUS).

(d) A bitumen layer (B) containing hydrophobic solids (HPS).

This preliminary separation scheme is illustrated on Figure 3-2. Appendix A lists the terms and definitions used to describe the fractions. Table 3-1 lists some of their more important, defining properties and typical concentrations

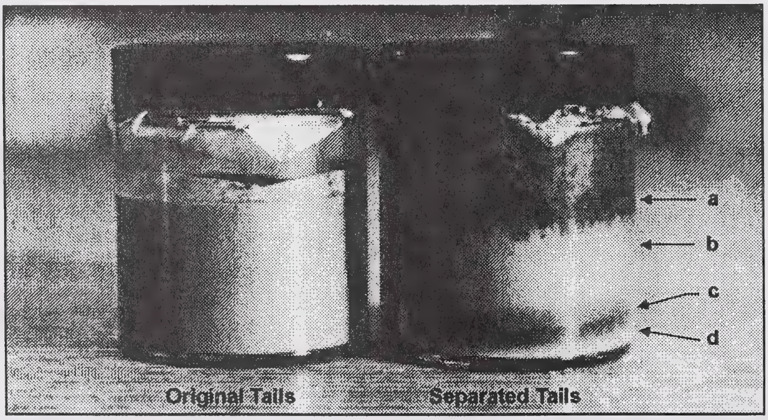


FIGURE 3-1. Separation of bulk fine tails by agitation and centrifugation: (a) bitumen layer and hydrophobic solids, (b) total ultra-fines suspension, (c) organic rich solids, (d) residual solids.

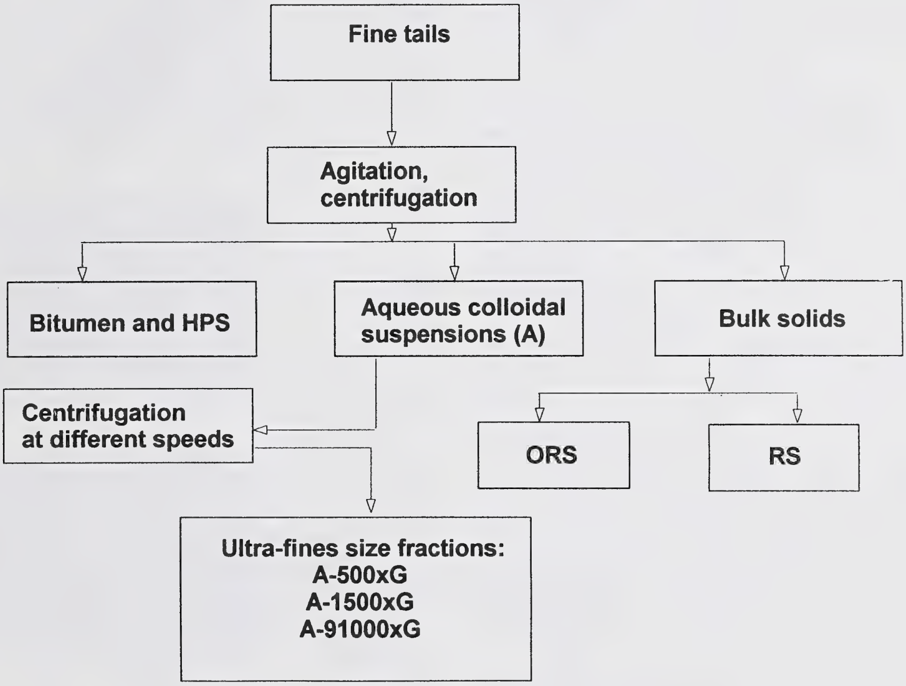


FIGURE 3-2. Separation scheme for fine tails.

TABLE 3-1. Amounts and properties of fine tailings components.

	HPS	HUS	BUS	ORS	RS
Concn. in MFT (w/w %)	1.5–2.5	2.7–3.2	0.8–1.3	1.5–2.5	20–25
Bulk composition (w/w %)					
IOCC	20–30	0.8–1.3	12.2–13.9	12–14	0.3–2.0
Carbonate	0.3	< 0.2	0.2–0.4	0.4–2.0	0.0–0.3
Fe	8–16	1.4–2.1	2.6–3.7	2–9	1–2
Ti	5–15	< 0.3	0.5	3	0.4
Kaolinite	NA	30–50	20–40	5–10	10–40
Mica	NA	25–40	10–25	10–25	10–25
Quartz	ND	ND	ND	5–50	85–90
Surface carbon (atomic %)	NM	< 4	>40	NM	NM
Particle size (μm)	NM	0.05–0.3	0.05–0.3	2–45	2–45

NA = Not Applicable, ND = Not Detected, NM = Not Measured found in Syncrude and Suncor fine tailings. Figure 3-3 shows electron micrographs, either scanning or transmission, of the different solids fractions. A number of conventional techniques have been used to characterize these tailings fractions.

3.2.1.1 *Bitumen Layer (B, HPS)*

The solids separated in this layer have a composition similar to low rank coals, such as lignite [11]. Organic carbon contents from 20 to 50 weight percentage and titanium concentrations up to 14 weight percentage are found in this fraction [12]. These solids interact strongly with bitumen and are never found as a separate component; they do not appear to play any role in tailings structure formation. In higher concentrations they may adversely affect the separation of bitumen by increasing its density.

3.2.1.2 *Ultra-fines Suspension Layer (A, HUS, BUS)*

There is an increased propensity of ultra-fines to form gels as their particle size decreases. It is therefore useful to determine the particle size distribution of these solids; in particular, the material less than 0.1 μm is most active. Before sizing, the ultra-fines must be deflocculated by removal of dissolved salts from the pond water. This is achieved by substituting distilled water for pond water in a series of centrifugation and wash steps. This process of ultra-fines separation and pore water replacement is completely reversible [13]. Particle size fractionation is achieved by sequential centrifugation of deflocculated suspensions at different speeds. Table 3-2 shows results for a typical fine tailings sample.

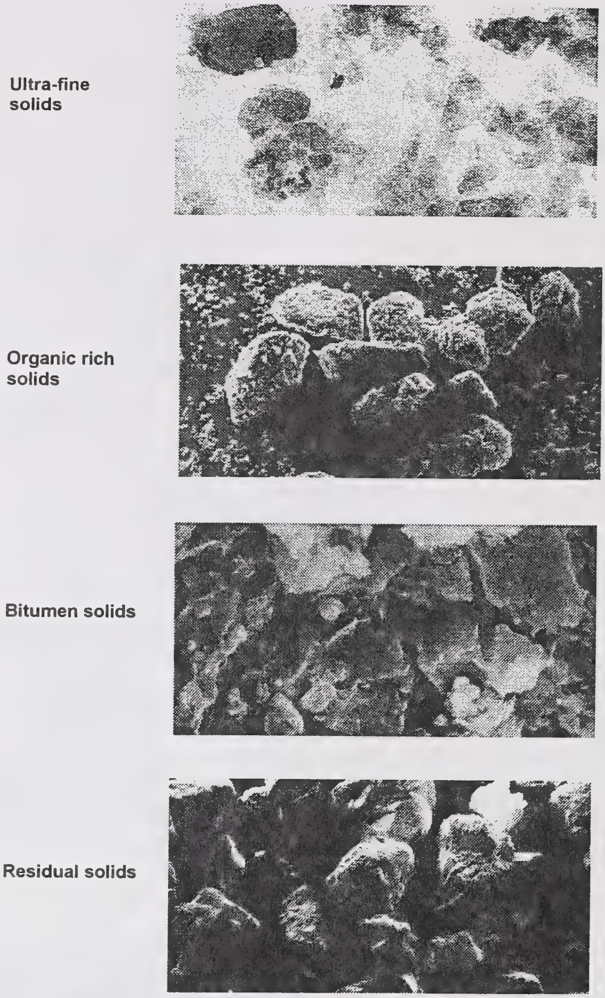


FIGURE 3-3. Scanning electron micrographs of fine tailings fractions.

Ultra-fines are clay particles in the size range 20 to 300 nm; the predominant minerals are kaolinite and mica [14]. These ultra-fine particles alone are the predominant water binding components in fine tailings. The particles are plate-

TABLE 3-2. Distribution and properties of ultra-fines size fractions.

Fraction	Average particle size (nm)	Distribution of solids[1] (w/w %)	Insoluble organic (w/w %)	Tiron extractables[2] carbon (w/w %)
1	400	35.0	0.5	2.9
2	200	25.0	0.9	10.2
3	100	22.4	1.0	N/A[3]
4	50	17.6	2.0	29.0

1. w/w percentage of total colloidal solids (bitumen free, dry basis)
2. Equivalent to non-crystalline content
3. Not available

like, as illustrated in the transmission electron micrograph (see Figure 3-3); the morphology is hexagonal to irregular. An interesting feature is that the clays are delaminated with an aspect ratio that often exceeds 30:1. Single layer clays are common in the finest size fractions [2]. The clays are X-ray amorphous but crystalline by electron diffraction [14]; treatment with Tiron, to dissolve amorphous material, also indicates a greater degree of crystallinity than observed by X-ray diffraction. The Si to Al ratio of 1.4:1 is higher than the theoretical value of 1:1 for kaolinite. It has been hypothesized that ultra-fines are composed of the mineral beidelite [3] or kaolinite from which Al atoms have been selectively dissolved from the exposed platelet edges. In either case the particles are highly charged [15] and hence very "active."

When toluene is used to remove residual bitumen from aqueous ultra-fines suspensions it causes the separation of a stable emulsion. This emulsion forms a separate layer or collects as a thick, cohesive "skin" at the interface between the solvent and the aqueous suspension layers. Further investigation revealed that the interface layer was composed of ultra-fine solids with biwettable surface characteristics. Stabilization of emulsions by fine solids of this type has been extensively reported in the literature [16]. These solids have been designated biwetted ultra-fine solids (BUS) while those solids with hydrophilic surface characteristics, remaining in suspension, have been designated as hydrophilic, ultra-fine solids (HUS). Contrary to Yong and Sethi's [1] prediction no amorphous iron oxide has been detected in any of the ultra-fine fractions.

The biwettable fraction represents about 20 weight percentage of the total ultra-fine solids. The material is associated with significant amounts of organic matter, strongly bound to the clay platelet surfaces (SOM). The characterization of this organic material has been described in great detail elsewhere [17–22]. In general terms the organic matter comprises elements of natural humic material and the more polar components of bitumen, such as resins and asphaltenes.

Tests have shown [23] that the adsorbed organic material affects particle behavior by altering the surface characteristics to make them less water wettable. Surface analysis by XPS shows that carbon accounts for about 40 atomic percentage of the surfaces for biwetted particles while for hydrophilic ultra-fines the value is less than 4 atomic percentage. XPS cannot distinguish between organic material present as either a uniform coating or as isolated patches.

3.2.1.3 *Organic Rich Solids (ORS)*

In the preliminary separation this material sometimes settles out as a layer of dark colored solids. Normally, the solids are found between the suspended ultra-fines and the clean quartz and clay layer at the bottom of the sample container. The solids in this fraction are relatively coarse with a size range of 0.5 to 45 μm. The particles are accretions of quartz, kaolinite, mica, heavy metal minerals, iron oxide, and siderite, bound together by organic matter. Removal of the organic matter by low temperature plasma oxidation results in the disaggregation of the composites into the individual mineral constituents [24]. The iron component is soluble in hydrochloric acid, indicating an amorphous nature. Although the ORS comprises less than 2 weight percentage of the total fine tailings solids it contributes 30 to 65 weight percentage of the total iron content. However, the amorphous iron is not a colloidal solid as required by Yong and Sethi's model. In fact his fraction corresponds to the "iron-organic complex," also proposed as a major contributor to tailings stability. The ORS fraction can participate in tailings formation, through the development of secondary structures [25]. However, measurements made on separated material show that, even under optimum conditions, it accounts for less than 10 weight percentage of the total water binding capacity of the tailings. Thus, there is some basis for use of amorphous iron content as a predictor for tailings volume. However, the correlation will be rather weak in many cases.

3.2.1.4 *Residual Solids (RS)*

These solids comprise about 90 weight percentage of the fine tailings solids and range in particle size from 0.3 to about 45 μm. Mean particle diameters are 8.1 μm and 24.2 μm for Syncrude and Suncor fine tailings, respectively. The major minerals are quartz, feldspar, and kaolinite. A scanning electron micrograph of this fraction (see Figure 3-3), shows rounded, fairly smooth, coarse grains. These are water wet and are associated with less than 2 weight percentage of strongly bound organic matter (SOM). In the fine tailings these solids do not contribute significantly to structure formation but merely occupy space within the gel system.

3.2.2 Ultra-fines in Oil Sands Ores

Having established that the basic gelation properties of fine tailings can be attributed to specifically identifiable species the next step was to determine the origin of these materials. An important question is whether the active tailings formers are present in the oil sands itself or are produced as a result of the treatment conditions used during the CHWE process. Direct application of the CWAT to oil sands ores demonstrates significant variations in the responses for different ore facies. In particular, marine ores do not produce ultra-fines suspension or free bitumen layers. The latter component is distributed throughout the sedimented solids layer. Also, although the ultra-fines themselves remain strongly aggregated they still separate from the bulk solids as a distinct layer on top of the coarser sedimented solids. For marine ores, the tailings water layer is usually completely free of solids.

To disperse strongly aggregated ultra-fines from marine ores a strong reagent, such as sodium pyrophosphate, is needed. The modified test for ore fractionation therefore includes an additional step in which the aggregated ultra-fines are treated repeatedly with fresh aliquots of sodium pyrophosphate solution. This information can be used to calculate a Dispersion Index (DI).

Values of DI range from 0 percent, for strongly aggregated ultra-fines, to 100 percent, for easily dispersible ultra-fines. Typically, marine ores contain less easily dispersible ultra-fines than estuarine ores. Although ultra-fines dispersability does not affect the final volume of MFT produced it does provide information on the rate of water released during settling to the ultimate tailings volume.

3.3 Water Chemistry Effects

3.3.1 Electrolyte Type and Concentration

The concentrations and type of electrolytes, in both formation (connate) water and pond pore water, play an important role in the behavior of ultra-fines. Ions may have either a dispersing or aggregating effect on the interactions between colloidal particles. In the original ore, electrolyte concentrations are high and the ultra-fines are present in a highly flocculated state. The presence of divalent cations in the formation water significantly reduces the dispersability of the ultra-fines aggregates. For example in marine facies aggregate strength is increased through the binding action of inorganic and organic compounds precipitated in the presence of calcium and magnesium.

During ore processing ultra-fines present in the oil sands ore are mobilized. At this point the concentrations of different ionic species in the tailings pond dominate the kinetics of ultra-fines aggregation. In turn, aggregation kinetics controls the rate of water release during the formation of mature fine tailings.

3.3.2 Oil Sands Facies

A preliminary comparison of formation water chemistry has been made between several samples of marine and estuarine oil sands; a complete description of the samples tested has been published elsewhere [26]. The major difference between the two ore types is the higher concentration of calcium and magnesium sulphates in the marine ores. A significant effect of this difference is that formation water pH and amount of water soluble organic matter are generally lower in marine ores than in estuarine ores [27].

It has been hypothesized that these observations explain the strong aggregation of ultra-fines in marine compared to estuarine ores. The presence of relatively low amounts of dissolved calcium ion in marine ores is believed to be an indicator that other soluble species, usually present in formation water, have been precipitated as the calcium salt. These would include calcium carbonate, calcium sulphate (gypsum), and calcium naphthenates. Such precipitates could act as a binder to reinforce the junction points between ultra-fine particles in the flocs. Because of these reactions marine ores contain strong flocs that produce less dispersed ultra-fines than estuarine ores when processed by the CHWE process.

While it is well known that calcium ions in pond water can have a beneficial impact on tailings behavior [28] excess calcium in the process recycle stream may have adverse effects on bitumen recovery [29]. Consequently, it is important to determine the response of different oil sands ores to recycle water containing different concentrations of calcium ions.

3.4 Discussion

The high water holding capacity of oil sands fine tailings is attributed to the presence of a three-dimensional gel network structure. This network is formed by ultra-fine solids, of colloidal dimensions, in the presence of electrolytes with a predominantly aggregating influence. Other tailings components, such as coarser solids and free or emulsified bitumen, are trapped in the network as it forms. These less active components may, or may not, contribute to the strength of the structure. During research on ultra-fines solids it has become apparent that the information developed allows conclusions to be drawn concerning several aspects of oil sands extraction:

- Tailings formation and volume prediction.
- Comparison of process performance, particularly the influence of process water chemistry on tailings formation and behavior.
- Reserve evaluation.

3.4.1 Ultra-fines Structure

3.4.1.1 *Sol-Gel Kinetics*

Visual observation of ultra-fines suspensions in electrolyte solutions, over time, gives the impression of progressively increasing viscosity until a stiff gel is formed.

The time for gelation depends on the initial concentration and particle size of the ultra-fines as well as the electrolyte type and concentration. These gels are thixotropic, meaning that their structure can be easily broken by application of shear stress. Nuclear Resonance Spectroscopy (NMR) provides a useful technique to provide information on gel structure and the sol to gel transition, that is the change from dispersed to flocculated particles [30]. This process can be monitored by means of ^{2}H or ^{17}O NMR spectra.

Figure 3-4 illustrates the spectra obtained using different sample handling protocols. Curves (a) are the spectra for an ultra-fines suspension allowed to gel before insertion into the NMR's strong magnetic field. Very little fine structure can be seen in either the ^{2}H or ^{17}O spectra and no changes occur when the sample is left in the magnet for several days. When this sample is shaken until it becomes free-flowing and replaced in the instrument the spectra produced are illustrated by curves (b) on Figure 3-4. The spectra now show the peak splitting detail characteristic of well dispersed particle suspensions. If the sample is left in the field while gel formation takes place some subtle changes are observed (curve (c)). While the peak splitting is invariant there is a change in line width of the doublet components, indicating a different relaxation mechanism [30]. These results confirm that gels can be produced in both orientated as well as random configurations. The orientated form is created by leaving a freshly shaken sample in the magnetic field to give the characteristic spectral signature illustrated by curves (b).

Apparently the gel structure can form from the sol without loss of orientated surface. This is an important observation as it precludes the formation of cubic structures, such as the classical cardhouse [31]. Systems are favored which feature ribbons or sheets with the platelets in edge to edge or face to face arrangements (see Figure 3-5). Macroscopic evidence of such structures can be seen in freeze fractured samples, viewed by a scanning electron microscope [32] and in sediments subjected to ultra-centrifugation at 366 kxG. In the presence of ribbon or sheet structures one would expect regions where the aggregated platelets have a common orientation with respect to the magnetic field; these regions may be termed domains. Again, similar features have been observed in photomicrographs of freeze-fractured tailings samples mixed at various intensities before freezing [32].

A study of flocculation kinetics, in dilute suspensions (0.06 volume percentage), of ultra-fines by photo correlation spectroscopy demonstrates that particle interaction depends, to a large extent, on water chemistry. This interaction between ultra-fines and dissolved salts, as illustrated on Figure 3-6, is

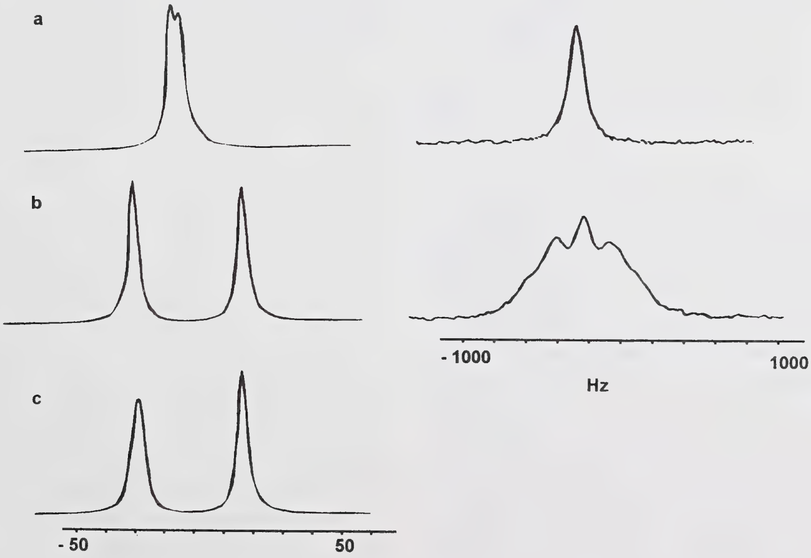
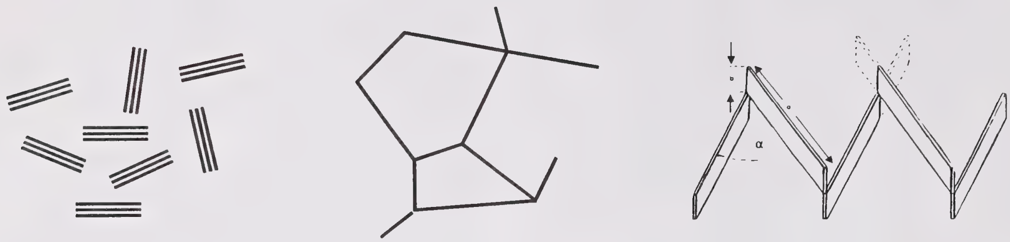


FIGURE 3-4. ^{2}H and ^{17}O NMR spectra of 5% ultra-fines sample after standing for one week (curves a) and of the same sample after shaking (curves b). ^{2}H NMR spectra of the sample for curves b after a residence time of two days in the magnetic field (curve c).



Face-to-face

Edge-to-edge

Ribbon-like

FIGURE 3-5. Configuration for edge-edge and face-face arrangements for clay platelets.

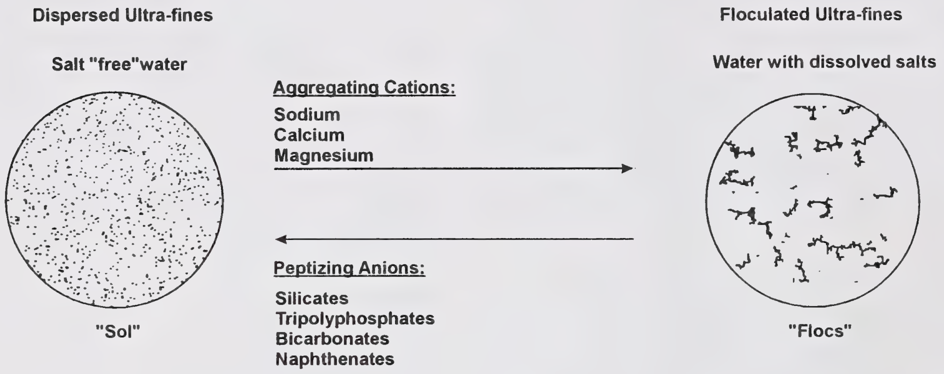


FIGURE 3-6. Effect of water chemistry on ultra-fines aggregation.

completely reversible. In distilled water there is little or no aggregation and the particles form a stable suspension or sol. Particle flocculation is initiated by the addition of cations; sodium ions are the dominant aggregating species in pond water.

Figure 3-7 shows plots of ultra-fines cluster size *versus* time for several concentrations of sodium chloride. At low salt concentrations ($\leq$5 mM) particle repulsion forces are of the same magnitude as their kinetic energy. Aggregation is limited by the need to overcome the repulsion barrier, nevertheless a slow, but exponential growth in floc size occurs (curve 1). This regime corresponds to reaction limited aggregation and is characterized by a collision sticking probability of less than unity. An increase in salt concentration to 10 mM further decreases the repulsive energy barrier and there is a corresponding increase in aggregation rate (see curve 2).

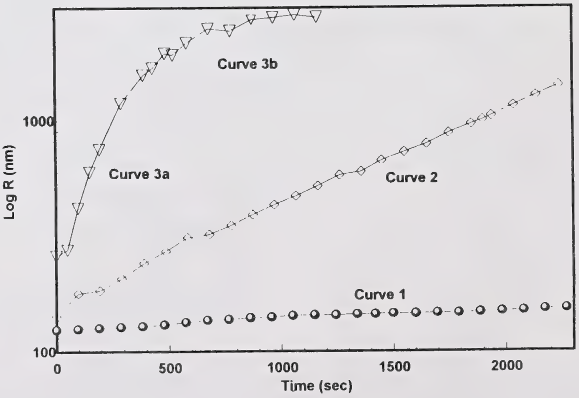


FIGURE 3-7. Mean floc radius *versus* time for three sodium chloride concentrations.

At a salt concentration of 20 mM two different flocculation regimes become apparent. Initially aggregation is reaction limited and there is little resistance to interparticle interaction and exponential floc growth occurs (curve 3(a)). As the flocs grow their numbers decrease, as does the probability of collisions between them. The flocculation process eventually enters a diffusion limited region (curve (b)). These two regimes are common for colloidal systems [33–35]. A feature of both regimes is that they produce open fractal clusters in which packing density, as measured by fractal dimension, decreases with increased cluster size. In contrast, non-fractal clusters are more compact and exhibit a uniform porosity [36]. An interesting feature is that the fractal flocs become more dense after being subjected to agitation. This may be an important consideration in oil sands processing where tailings are pumped over considerable distances. Floc densification is an important factor in tailings thickening.

3.4.1.2 *Ultra-fines Settling and Tailings Formation*

The same dilute suspensions, used in the kinetic study, were allowed to settle over an extended period. The behavior of the suspensions was monitored by following the interface between the suspended solids and the clear supernatant water layer. Concentration ranges of electrolyte, either sodium chloride and/or sodium bicarbonate, were selected to cover the reaction and diffusion limited flocculation regimes. A blank sample containing no salts was also used for comparison. Below 50 mM of sodium bicarbonate all the suspensions remained stable, demonstrating the dispersing action of bicarbonate ions. At concentrations of sodium chloride below 5 mM aggregation is slow, whereas at concentrations ≥20 mM there is fast, diffusion limited aggregation. The differences in behavior between suspensions in either of the two electrolytes diminished markedly as their concentrations were increased; at concentrations of 100 mM, and higher, the two salts had almost the same effect on flocculation. At the higher concentrations of salts the aggregating action of sodium ions completely dominates the dispersing characteristics of bicarbonate ions.

At sodium chloride concentrations ≤5 mM ultra-fines flocculation occurs in the reaction limited region and differential settling behavior is observed [37]. Those particles that have a lower resistance to flocculation aggregate and settle first. The remaining solids stay in suspension above the fast settling layer. Differential settling is expected to create conditions favoring the formation of segregating tailings. With higher concentrations of suitable electrolytes diffusion limited aggregation results in rapid initial ultra-fines flocculation followed by fast settling. These conditions favor entrapment of coarser solids in the flocs, leading to non-segregating tailings. These observations are illustrated graphically on Figure 3-8 and visually by the photograph on Figure 3-9. Regardless of the mode of formation, ultra-fine flocs settle freely until a concentration of 1 to 1.4 volume percentage solids is reached. At this point the interfloc distances are small and the flocs begin to interact. Ultimately, a giant spanning cluster is formed at the critical gel concentration for the ultra-fines sample under study [26].

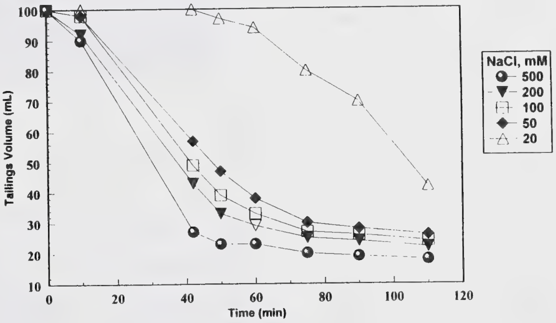


FIGURE 3-8. Effect of sodium chloride concentration on rate of settling and water release from ultra-fines suspensions.

It is important to note that the final settled ultra-fines content, at the gel point, is nearly the same regardless of their concentration in the original suspension. The time to reach the gel point, and the ability to entrap coarser solids, depends on the water chemistry, the original concentration of ultra-fines, and their degree of dispersion (Dispersion Index). Higher ultra-fines content and electrolyte concentrations produce gels more rapidly. In such situations the ability to capture coarser solids is enhanced. Analyses of samples from both Syncrude and Suncor tailings ponds always yield a concentration of ultra-fines in the range of 1 to 1.4 volume, or 3 to 4 weight percentage.

The rate of water release during formation of mature fine tailings is also dependent on ultra-fines concentration together with electrolyte type and concentration in the pond. However, if the ultra-fines content is initially greater than the critical gel concentration and an appropriate concentration of aggregating ions is present, very little, if any, settling will occur. That is, the tailings will totally occupy the volume into which they are initially placed and virtually no water release will occur at all.

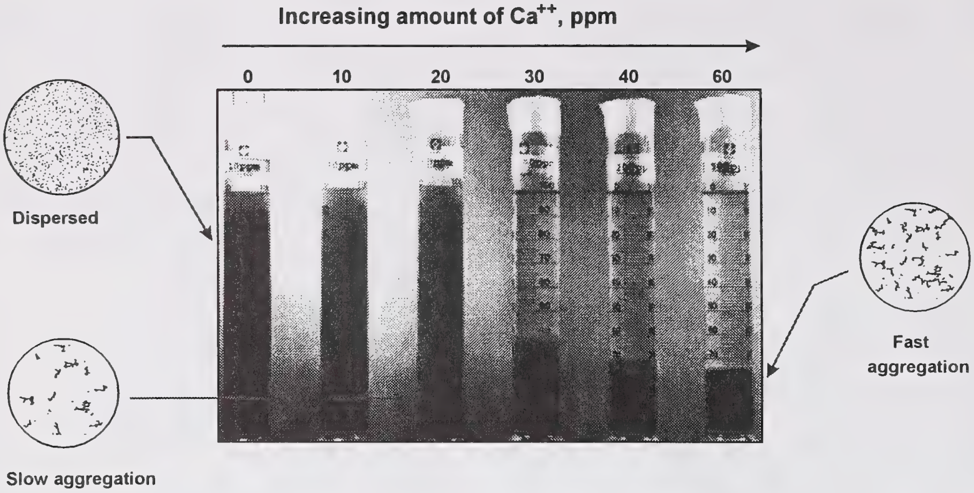


FIGURE 3-9. Visual effect of slow and fast aggregation regimes.

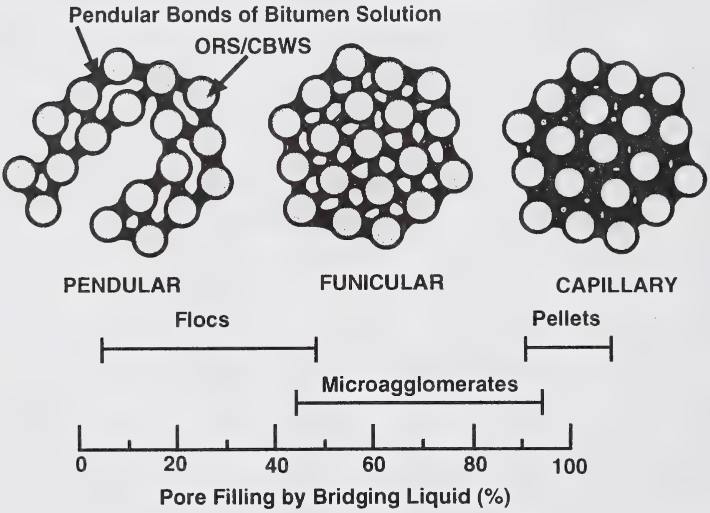


FIGURE 3-10. Agglomerate forms.

3.4.2 Secondary Structures

The organic rich solids (ORS) represent 2 to 3 weight percentage of the total MFT. Like the biwetted ultra-fines fraction these solids exhibit an intermediate surface wettability; this property allows them to interact with both water and oil. Similar properties are exhibited by low rank coals. In the presence of two immiscible liquids such solids are not completely wetted by either liquid.

It is well known that when a particulate solid is mixed in the presence of two immiscible liquids the particles will agglomerate. This occurs as a result of interaction between layers of the liquid preferentially adsorbed on the particle surfaces [38]. This so-called bridging liquid is usually present as the minor liquid component. When the solid surfaces are incompletely wetted by the bridging liquid the number of bonding points between particles is limited and only voluminous flocs can be formed. Under these circumstances the liquid bridges between particles are pendular in nature (see Figure 3-10).

Flocculation tests have been conducted with samples of ORS selectively separated from MFT [25]. The solids were suspended in pond water and mixed with various amounts of a bitumen solution in naphtha. The mixtures were placed in measuring cylinders and the final, equilibrium volume of sediment measured. Figure 3-11 shows a plot of normalized final sediment volume *versus* amount of bridging liquid added. The maximum volume of bitumen solution used in these tests corresponded to a bitumen content of about 3 weight percentage of MFT; this is typical of samples taken from Suncor's pond 1. Samples of ORS from both Syncrude and Suncor were tested; both gave similar results. In each case the sediment volumes increased gradually with amount of bridging liquid added, finally leveling off at a value close to three times the original sediment volume of the solids.

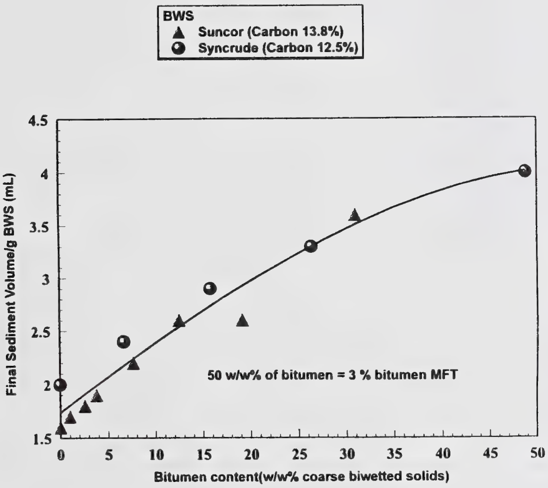


FIGURE 3-11. Sediment volume of biwetted, coarse solids *versus* added bitumen solution.

A simple calculation shows that the amount of ORS, typically found in tailings, has the capacity to hold 8 to 10 weight percentage of the total water present in the MFT. This is a relatively minor contribution compared to that associated with ultra-fines. However, the capillary forces, associated with pendular liquid bonds, are very strong. The effect of these flocs on tailings viscosity and load bearing capacity may be much greater than their contribution to water holding capacity would indicate.

Such flocs will only form under conditions where ORS are contacted with low viscosity bitumen solutions under fairly intense mixing conditions. Centrifugal pumps are quite capable of providing adequate mixing intensity and retention time. The ORS fraction is also associated with a major part of the amorphous iron present in fine tailings. Consequently, there will be some correlation between iron content and the propensity of ores to form tailings. Thus, there is some basis for amorphous iron content of tailings being used as an indicator for tailings volume prediction as proposed by the proponents of both the "iron-organic complex" and colloidal, amorphous iron models. However, very little iron is associated with ultra-fines and given the wide range of iron contents found in the ORS fraction it is expected that the correlation of iron content with tailings volume will be weak, at best.

The fine size of ultra-fines implies that they have a high surface area. Tailings prediction models that rely on surface area determination, such as methylene blue adsorption, should therefore be good indicators for tailings volume; in reality the method has not proved to be reliable. This is not surprising when one considers that adsorption measurements are made on whole tailings. In these circumstances there is a strong probability of interference from the high concentration of clay particles other than ultra-fines. More reliable results may be possible by using the methylene blue test on the crude ultra-fine suspension separated in the CWAT method.

3.4.3 Tailings Volume Prediction

Results show that the ultra-fines content of an oil sands ore, as determined by the methods described earlier, is a primary predictor for the formation of mature fine tailings. A combination of ultra-fines content and dispersability index provides information on the rates of tailings formation and water release. Although different ore facies contain ultra-fines with different degrees of dispersability the final volume of tailings is always governed by the absolute value of the critical gel concentration (CGC) of the ultra-fines fraction. The time taken to reach the CGC depends on the electrolyte concentration and degree of dispersability of the ultra-fines. For example, strongly aggregated ultra-fines, such as those from marine ores, settle much faster than do the more easily dispersible ultra-fines from estuarine ores.

A simple calculation demonstrates that the amount of ultra-fines in MFT will account for almost all of its water holding capacity. For this determination it is necessary to know the weight or volume fraction of ultra-fines in the water phase of the MFT and their critical gel concentration. Also, it is possible to demonstrate that the water holding capacity for different ultra-fine size fractions is approximately additive.

Eq. (1) is used, where (α is the percentage of tailings water bound by ultra-fines, W_{UF} is the weight fraction of ultra-fines with respect to the water content of mature fine tailings, and W_{CGC} is the measured weight fraction of ultra-fines at the critical gel concentration.

$$(1)\quad \alpha = \left[\frac{W_{UF}\text{ in MFT water}}{W_{CGC}}\right] \times 100$$

W_{CGC} can be determined for the whole ultra-fines fraction or for several individual size fractions. Where the latter information is available the additivity of the property can be determined. Sample data for these calculations is given in Table 3-3. A summary of the calculations combining the data from Table 3-3 with Eq. (1) are given in Table 3-4. As can be seen 3 to 4 weight percentage of ultra-fines is capable of binding almost all the water found in mature fine tailings. The sum of water binding capacities for sized ultra-fines fractions is reasonably close to the value calculated for the ultra-fines composite layer from the original separation. These calculations compare favorably with those made by Yong and Sethi [1], who assumed that a more complex suite of colloidal sized minerals was responsible for the water holding capacity of fine tailings.

Although the amount of water associated with the ultra-fines solids is very large it is not all tightly held. Calorimetric measurements show that only 7 weight percentage of the total water is bound closely at particle surfaces [39]. The remaining water is free but is constrained by the loose, ultra-fines gel structure. There is plenty of space within the gel network to accommodate entrapped particles that do not necessarily contribute to its structure. However, incorporation of coarser particles into the tailings will obviously result in a higher solids loading. The capacity of fine tailings to capture coarser solids depends on its gel strength, which in turn largely depends on water chemistry.

After mixing with either calcium fortified (simulated river water) or distilled water a series of estuarine and marine ores, see Table 3-5 for descriptions, were placed in cylinders

TABLE 3-3. Composition and properties of Syncrude and Suncor MFT.

Components	Suncor (w/w %)		Syncrude (w/w %)	
Total solids	26.3		26.0	
Bitumen	1.9		1.0	
Water	71.8		73.0	
Ultra-fines fractions:	UF (w/w)	CGC (w/w)	UF (w/w)	CGC (w/w)
Total	3.6	6.2	3.8	5.5
200 to 300 nm	1.6	8.0	1.9	6.6
100 to 200 nm	1.5	5.4	1.4	5.3
<100 nm	0.5	2.20.5	2.0	

TABLE 3-4. Water holding capacity of ultra-fines from Syncrude and Suncor MFT.

Ultra-fines fraction	Percentage of total tailings water bound by ultra-fines	
	Suncor	Syncrude
300 nm	27.8	39.4
100 to 300 nm	38.7	36.2
< 100 nm	31.6	34.3
Sum for fractions	98.1	109.9
Total ultra-fines fraction	80.9	94.6

TABLE 3-5. Description of oil sands facies used in tailings formation test.

Oil sands origin	Sample #	Facies	Description
Marine (M)	M-I	26	Dark, black carbonaceous clays in a relatively clean coarse sand from the top lift of ore in the north side of the Auxiliary Pit.
	M-II	16	Bioturbated oil sands on top lift on north face of the Auxiliary Pit. Mottled appearance due to discontinuous wavy stringers of dark gray to black clay (shaley). The entire unit is highly burrowed. Low bitumen saturation in this waste unit (?).
	M-III	18	Massive rich oil sands from the upper ore lift on the north west end of Auxiliary Pit.
Estuarine (E)	E-I	22	Silty unit approximately 20 to 30 cm thick that underlies Facies 11 unit (sample #3). Numerous white sandy stringers exhibiting burrow mottling.
	E-II	6	Silt-clay clasts in a Breccia in low grade ore located in the south east corner of the Auxiliary Pit. Thinly laminated and interbedded silts and waxy clays. Tidal flat sediments with thin discontinuous stringers of clean white sand. Sand stringers appear to occupy irregular shaped worm burrows as well as comprising thin flaser laminations.
	E-III	21	Very well laminated sediment with much carbonaceous laminae.
	E-IV	8	Well burrowed sediments located on the north wall of the Auxiliary Pit at about 260 m elevation.
	E-V	16	Thick unit with high bitumen saturation located at approximately 260 m elevation at the west side of the Auxiliary Pit. Minor clay breccia clasts in the rich oil sands unit near its base. These clay clasts do not comprise part of the sample.

to settle freely. Coarse sand, equivalent to primary tailings, settled out almost immediately. An ultra-fines layer, the precursor of MFT, formed above the coarse sediment, gradually settling out over a much longer time. The volume of this ultra-fines layer was monitored until it reached an approximately constant value. The time taken to reach the final equilibrium volume varied, depending on the ore and the suspending medium. Typically, the marine ores and all samples in contact with calcium fortified water were observed to give fast settling. A plot of ultra-fines concentration *versus* final volumes of the ultra-fines layers for ores in both distilled and calcium fortified water is shown on Figure 3-12. In terms of ultra-fines aggregation, the water chemistries selected represent the extremes for possible treatment conditions. Three important conclusions are apparent from this plot:

1. Independent of ore type and degree of ultra-fines dispersion there is an approximate linear correlation between the amount of ultra-fines in each individual ore and the final volume of sediment.

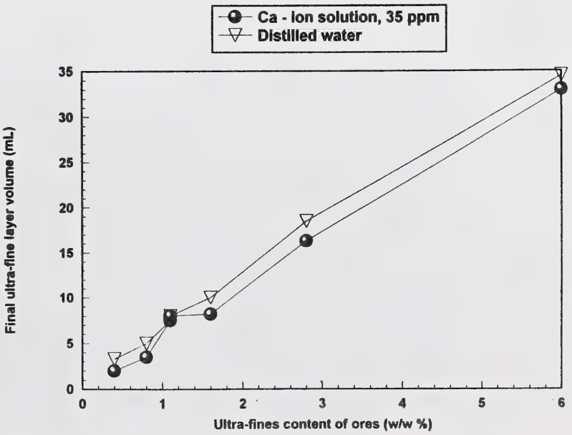


FIGURE 3-12. Final volumes of fine tailings from a series of ores processed with either distilled water or calcium fortified water.

2. The final volumes of the ultra-fines layers are about the same in distilled and calcium fortified water. In each case the distilled water samples have a slightly higher volume because of the more dispersed state of their ultra-fines component. Ultimately, the two curves are expected to converge.
3. The time taken to reach the final volume was much less for those samples in Ca^{2+} fortified water.

These observations are important in that they demonstrate that knowledge of the ultra-fines content of an ore is a good predictor for fine tailings production. Also, water chemistry and degree of ultra-fines dispersability are a reliable indicator for rate of water release. Both of these parameters are crucial to the mine and tailings pond planning process.

3.4.4 Process Water

Typical samples from Syncrude and Suncor's tailings ponds have similar water chemistry. Sodium and bicarbonate ions are the predominant, active species. Although the existing pond water originated from local river sources, having a relatively high calcium content, continuous recycle has changed its make-up by augmentation of sodium, chloride, and bicarbonate ions. Calcium has become depleted by reaction with the bicarbonate ions and other species, such as naphthenates, to produce insoluble precipitates.

Alternative bitumen extraction methods, such as the OHWE process, use surface water that naturally contains, or is enriched with, calcium ions. The reject streams from these processes give faster settling, non-segregating tailings with sufficient strength to support sand surcharging as part of a tailings thickening scenario. Also, it is well known that marine ores produce fast settling tailings [40], again this is believed to be caused by a much higher bivalent ion content in these ores, compared to estuarine facies. As a result of these observations addition of calcium ions to process water is expected to produce more tractable fine tailings in terms of water release and strength. However, over-dosing with calcium can be detrimental to bitumen recovery [41]. Consequently, a means of estimating the appropriate level of chemical addition is required.

Several ores, having widely different properties, were selected for use in a series of tests to determine optimum calcium ion addition. The objective was to convert slow settling conventional tailings to a fast settling type, typical of tailings from marine ores. A summary of the ore properties is given in Table 3-5. Each ore was then treated with progressively higher concentrations of calcium ions in the water added as part of the CWAT. The photographs on Figure 3-13 show typical results for the Syncrude ore used in the OHWE-CHWE pilot plant comparison tests. For this ore water containing 75 ppm of calcium ions produced a marine ore type tailings. Other ores require different calcium ion dosages [42].

The optimum calcium ion concentration does not correlate directly with any single ore property. Its value is probably determined by a complex interaction between the calcium saturation concentration of the ore and dissolved carbon forms, both organic and inorganic. However, the procedure does provide a method for estimating calcium ion addition to process water for prevention of over-dosing.

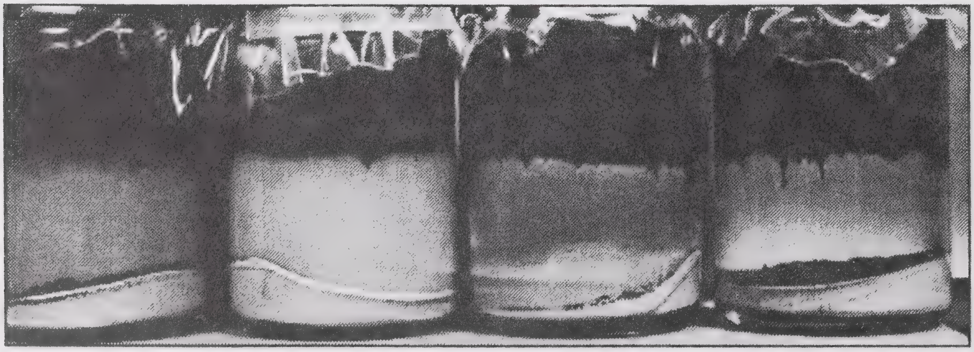


FIGURE 3-13. Conversion of CHWE tailings to OHWE tailings by Ca^{+2} addition.

Another process option is to adopt the reverse procedure and produce a completely deflocculated tailings by using a strong dispersing agent as a process aid [43]. Potential candidates for this purpose are sodium silicate or sodium pyrophosphate. Dispersed ultra-fines do not exhibit the structure forming properties found in conventional fine tailings. Consequently, it becomes possible to recycle process water with a high solids content without inhibiting bitumen separation because of high viscosity in the PSV middlings. This approach has been successfully tested in two series of pilot tests [10].

3.4.5 Chemical Treatment of Tailings

Over the years many attempts have been made to reduce tailings volume by chemical treatment. These include MFT treatment with lime and other inorganic flocculants, polyelectrolytes, pH control, and combinations of these process aids [44]. All of these approaches have been either technically or economically unsuccessful in increasing the final density of the tailings to an adequate level. Usually, chemical treatment of a tailings sample with about 30 w/w percentage solids produces a final solids content in the range of 36 to 40 w/w percentage. However, significant increases in dewatering rates and gel strength of the treated tailings do provide some benefits in terms of handling and final disposal. Best results have been obtained by acidifying to pH 4 and then neutralizing to pH 7.5 with lime [45].

The most active ultra-fine fraction (<0.3 μm) is always present in MFT at concentrations in the critical range of 3 to 4 weight percentage. This small amount of material controls the overall thickening of the tailings during chemical treatment. To determine directly the effect of chemical treatment on this fraction some test work was carried out on ultra-fines, separated according to the standard technique [46]. The sample used contained a concentration of 7.6 weight percentage of particles less than <0.5 μm in size. This concentration is close to the CGC for this size fraction.

Acidification over the pH range 1.9 to 8.4 and low speed centrifugation, to simulate several months of gravity settling [47], produced little if any thickening of the solids. However, subjecting the suspension to a single freeze-thaw cycle followed by centrifugation increased the solids concentration by a factor of 3.5 (25 to 27 weight percentage) over the pH range of 2 to 6.4. If the acidified suspensions are neutralized to pH 7.6 with lime before centrifugation the results were very similar, indicating no particular process benefit for this treatment, even after freeze-thaw. Suspensions of ultra-fines can also be compacted, without chemical treatment, to concentrations in the same range by ultra-centrifugation at 366 kxG. These results indicate that only powerful mechanical forces, such as those experienced during freezing and centrifugation, are capable of collapsing the ultra-fines structure.

3.4.6 Comparison with Similar Tailings

Other fine textured sludges are known to respond to mechanical handling methods that promote evaporative removal of water from layers of tailings spread over a wide area [48]. In Holland, dredged material from local harbors and waterways, is successfully treated by this approach. These methods have potential to be a cost effective tool in an integrated approach to tailings disposal. However, oil sands fine tailings have proven difficult to treat by these techniques. The weak gel structure of these tailings impedes the use of the mechanical farrowing equipment used to increase the surface area of the tailings layer for improved water evaporation. Also, the tailings readily form an impermeable surface crust that inhibits further water loss. The major difference between the two test materials was the greater degree of stability to mechanical stress exhibited by the dredger sludges. This allows the latter to be more easily worked by mechanical equipment.

To determine the reason for this difference a comparative study, based on application of the CWAT procedure, was carried out [49]. Results from these tests showed a similarity in ultra-fines content and particle size distribution for both suspensions. The most significant difference was in the amount and type of organic material associated with the ultra-fine solids fractions. Whereas only a portion of oil sands ultra-fines are associated with strongly bound organic matter, the dredger sludges contain large amounts of easily extractable (immature), humic matter. Also, the dredger sludges did not contain any biwettable ultra-fines.

Humic matter is a well known aggregating agent. Particle interactions occur through the so-called bridging flocculation mechanism [50]. This process occurs when a portion of an adsorbed humic molecule lies away from the surface as loops or tails that are able to adsorb onto the surfaces of other particles to produce linkages. If this difference in structure is the explanation for the distinct behavior for the two types of fine tailings it opens the intriguing possibility that MFT can be converted to a more tractable material. This could be achieved by treatment of oil sands fine tailings with an appropriate form of humic matter. Suitable material may be readily available locally from the muskeg stripped from the site prior to commencement of mining.

3.5 Conclusions

3.5.1 Economic Impacts

- A fundamental understanding of the nature of fine tailings has led to the realization that its production may be reduced simply by changing the conditioning agent used during bitumen extraction. This may be achieved by substituting a strong dispersing agent, such as sodium silicate, for the conventional sodium hydroxide. This simple change eliminates the flocculating propensity of ultra-fines and its concomitant adverse effect on slurry viscosity. Under these circumstances it becomes possible to recycle tailing's water with a high ultra-fines content. Compared to current disposal practices this process will result in a greater level of ultra-fines entrapment in the pores between coarser sand grains in the dykes and beaches.
- Development of a simple test procedure allows mature fine tailings and oil sands ores to be separated into several fractions each having its own particular characteristics. In its simplest form this test provides a quantitative measure of total ultra-fine content. This information is a better indicator for tailings production than the total fines (<45 μm) content of ores and is anticipated to be more reliable than the methylene blue adsorption test. More sophisticated developments provide information on ore processability and rate of water release from tailings. This test is expected to become the industry standard for evaluating oil sands ores.
- Breaking the ultra-fines structure allows the release of a number of solids fractions that may have some commercial value. These include: a gelling agent, a biwetted, ultra-fine fraction with powerful emulsifying properties, a heavy metal concentrate and kaolin clay with potential for use in coating applications [51].

3.5.2 Scientific and Engineering Impacts

- The most important scientific outcome of this research program is the recognition that the unusual properties of fine tailings are predominantly associated with the gelation characteristics of an ultra-fine, colloidal (<0.3 μm) solids' fraction. These plate-like particles are delaminated kaolinite and mica with hexagonal or irregular morphology. Ultra-fines exist in a naturally flocculated state as a result of the electrolytes present in formation and pond water. The concentration of ultra-fines in oil sands fine tailings is sufficient to account for more than 90 percent of its water holding capacity. In all tailings samples tested the ultra-fines are present at their critical gelation concentration of 3 to 4 w/w percentage.
- It has been demonstrated that ultra-fines solids originate in the parent oil sands ores. The degree of mobilization of these solids, during processing for bitumen extraction, depends on the ore facies and the process conditions. Typically, the dispersability of ultra-fines flocs is greater for estuarine than for marine ores.
- In terms of wettability ultra-fines exist primarily in two forms, namely hydrophilic and biwetted types. The difference between the two lies in the amount of organic matter bound to their surfaces. For the hydrophilic fraction surface coverage by organic carbon amounts to <4 atomic percentage while for biwetted material this increases to about 40 atomic percentage. In actual fact the wettability characteristic of the ultra-fines is a continuum and these numbers represent average values for the two extremes. The organic material is a mixture of humic matter and asphaltic components from bitumen strongly bound to the clay surfaces. The biwetted solids accelerate the gelation process and stabilize bitumen emulsions which, in turn, increase tailings viscosity.
- The stability and behavior of ultra-fines flocs also depend on the amounts and types of electrolytes in the process water. Consequently, a complete understanding of tailings behavior requires knowledge of ultra-fines content, the *in situ* environment in the parent ore, and process or pond water chemistry. An important observation is that ultra-fine particles obey all the basic laws of colloid chemistry.
- The major electrolytes affecting ultra-fines flocculation in pond water are sodium and bicarbonate ions; the former exerts an aggregating effect while the latter has a dispersing action. The final degree of ultra-fine flocculation results from an equilibrium between these opposing effects.
- Once released into the tailings' pond the ultra-fine flocs settle until they reach the critical concentration (3 to 4 w/w percentage) at which a space filling gel forms. The fully formed structure is capable of supporting some coarser particles.
- Contributions to tailings' structure have also been recognized from emulsions of bitumen and naphtha stabilized by biwetted ultra-fines and bitumen bonding of coarser biwetted solids. Both of these mechanisms result in thickening and strengthening of the basic ultra-fine gel structure.

3.6 Acknowledgments

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APPENDIX A

List of Abbreviations and Terms

A Aqueous suspension layer separated in the **CWAT** procedure. Contains all solids less than 0.5 μm in diameter; this fraction is considered to be colloidal in nature.

A-200, A-200xG

Fraction of suspended solids sedimented during centrifugation at 200xG.

AS, HUS

Hydrophilic (water wettable), ultra-fine particle suspension; that part of fraction **A** remaining after removal of **BUS**.

B Bitumen layer separated during the **CWAT** procedure.

BS, HPS, OPS

Relatively coarse solids separated with the bitumen layer during the **CWAT** procedure; particles have an organic matter content and exhibit hydrophobic (oil wettable) surface characteristics. This fraction contains the highest concentration of heavy metal minerals.

BUS, SAS, FBWS

Biwetted (both oil and water wettable), ultra-fine particle suspension separated from fraction **A** by emulsion flotation with an organic liquid; Surface Active Solids; Fine Bi-Wetted Solids.

CBWS, ORS

Coarse, Biwetted Solids; Organic Rich Solids. A fraction similar to **HPS** but containing lesser amounts of organic (humic and/or asphaltic) matter and heavy metal minerals.

CGC

Critical Gelation Concentration: the minimum concentration of colloidal solids, of a given size, required to form a stiff, thixotropic gel. **CGC** decreases as particle size becomes smaller.

CHWE

Clark Hot Water Extraction Process.

C_{on}, GOC

Gel Onset Concentration: the concentration of colloidal solids required to produce a noticeable thickening of an aqueous suspension of the solids.

CWAT

Cold Water Agitation Test procedure; method for separating colloidal solids from other fine tailings fractions.

GOT, t_{on}

Gel Onset Time: the time required for noticeable thickening of a suspension of ultra-fine solids to occur.

GP Gel Point is equivalent to the **GOP** or **GOT**

IOCC, IOM, SOM

Toluene Insoluble Organic Carbon Content; Insoluble Organic Matter; Strongly Bound Organic Matter. This material comprises humic and asphaltic type components that are difficult to displace from solids' surfaces.

MFT

Mature Fine Tails: tailings having a solids content of about 30 w/w percentage.

OHWE

OSLO Hot Water Extraction Process.

PSD Particle Size Distribution.

RS Residual Solids: the coarser fraction (>0.3 μm) of **MFT** that is settled out in the **CWAT** procedure. The fraction is relatively free of organic material and largely comprises quartz and kaolin minerals.

NOTE: the underlined abbreviations are the currently recommended designations.

4.0 FUNDAMENTAL PROPERTIES OF FINE TAILS

4.1 Introduction

Mature fine tailings (MFT) from the Clark hot water process have been thoroughly characterized by a wide variety of methods [1–7]. These characterizations have revealed that the MFT component interactions at the microscopic level determine the bulk geotechnical and physical properties of the mature fine tailings. Several models have been proposed over the years to describe these interactions. Work sponsored by the Fine Tailings Fundamentals Consortium has made significant progress in resolving two outstanding issues regarding the structure and properties of MFT: (1) the relative importance of mineral or organic fractions in determining MFT properties and (2) the gel *versus* floc (or aggregate) structure of MFT. Based on the most recent studies, mature fine tailings can be described as a suspension where the settled volumes and water holding capacities are determined by the mineral components rather than by the residual bitumen. The gel *versus* floc structure descriptions have been reconciled with a fractal description of MFT. In other words, the clay interactions or floc structuring is a continuum from the very smallest ultrafines to clay size (2 micron) and larger. For the large clay platelets, the settling is slowed by the viscosity of the suspension and this provides an environment where coarser particles can also slowly settle or orient into a particular flocculated or aggregated structure. Furthermore, the strength of this floc structure, and therefore the bulk properties of MFT, are a function of the chemistry of the suspension water and in particular, the pH. At the MFT pH of 8.4 (determined by the bicarbonate ion buffer), the flocculated clay structure is weakest. At higher or lower pH, the clays are flocculated in a different way and the MFT structure is strengthened [3].

Addition of divalent ions also strengthens the floc or aggregate structure and can result in a material that will support an internal surcharge of sand (nonsegregating) which enhances consolidation. Nonsegregating mixtures can be created by manipulating the concentration of divalent ions (typically *via* calcium addition from gypsum), or by a combination of pH changes (to move away from the pH determined by the bicarbonate buffer) and calcium addition (typically acidification to remove bicarbonate followed by calcium addition from lime to return to a near neutral pH).

The floc or aggregate structure can also be changed by freeze thaw strains which result in the physical collapse or compression of the floc or aggregate structure. The degree of structure collapse and the reduction in MFT volume with freeze thaw is enhanced by chemical treatment to change the floc structure before a freeze thaw cycle.

4.1.1 Mature Fine Tailings Properties and Structure

The water holding capacity of MFT, or the slow consolidation rate is governed by the surface properties of the minerals. The forces that affect colloidal particles in suspension and determine final settled volume, permeability, and strength of the material have four essential components: (1) electrostatic, (2) steric, (3) Van der Waals, and (4) hydration. In a complex suspension like mature fine tailings, quantifying the contributions of each of these forces can be difficult. Divalent ions such as calcium have a large effect on the the electrostatic component, resulting in rapid settling, but with limited decrease in the settled volume (see Figure 4-1). Similar behavior is observed with MFT that has had the bitumen component removed by solvent extraction; that is, rapid initial settling but to a similar final volume.

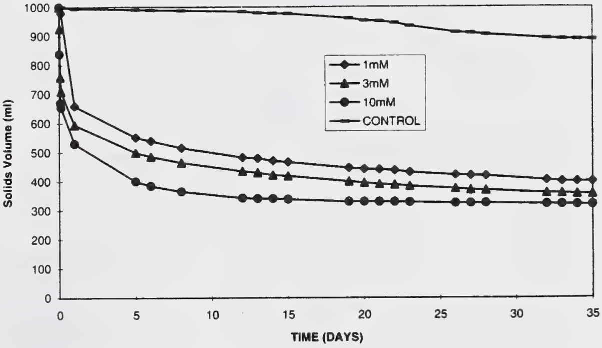
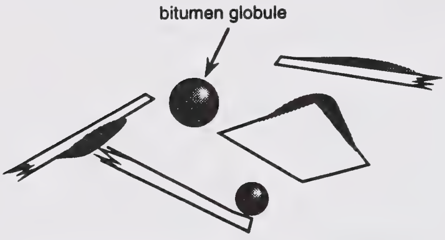


FIGURE 4-1. The settling of tailings clays as a function of added calcium.

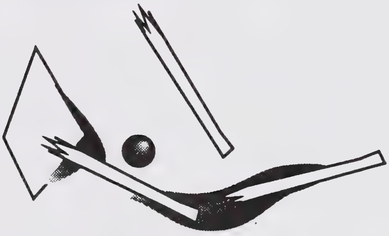
4.1.2 The Mineral *Versus* Organic Models of MFT

The low permeability and slow consolidation of the MFT is a function of how the bitumen, solids, and water interact. Ultimately, the solids component is the most important since the rate at which the solids settle and consolidate determines the capacity and lifetime of the containment ponds. The nature of the interaction between the solids is a function of the water chemistry and the solids' surface (the mineralogy or the potential for organic contamination). The bulk of the fine particles are kaolinite and illite clays, with some quartz and other components. Bitumen is also present in variable amounts. Two models of MFT structure naturally arise from these data: one based on the role of organic material, the other based on the mineral properties. Figure 4-2 shows the organic or bitumen model proposed by Scott *et al.* [6] and Figure 4-3 shows the mineral model proposed by Yong and Sethi [7]. Early work by the consortium concentrated on mineral/organic interactions and an organic based explanation for MFT properties. The dispersive nature of the Clark hot water extraction process and the release (in the caustic environment) of petroleum surfactant supported this approach. In addition, the mineral model proposed by Yong and Sethi required quantities of montmorrillonite and amorphous iron that are not present in all MFT samples.

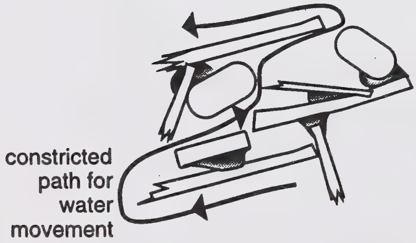
Studies of the electrophoretic properties [8–10] of MFT clays and characterization of hydrophilic fines from MFT [11–13] resulted in a shift in research to the mineral model of MFT stability. The electrokinetic behavior of kaolinite and illite clays in recycle water showed behavior quite different from kaolinite in pure water. This was initially interpreted as being due to organic components in the water interacting at the clay surface. Subsequent studies revealed that this behavior occurs in the presence of bicarbonate ions, on ideal clay suspensions with no organic contamination (see Figure 4-4).



(a) Bitumen adsorbed on particle surfaces



(b) Particle agglomeration



(c) Reduction of sludge permeability

FIGURE 4-2. The organic control of mature fine tailings properties (from Scott *et al.* [6]).

Kotlyar *et al.* separated a hydrophilic fines fraction (having little or no organic component from MFT that gelled at relatively low concentrations in suspension [11–15]. Further characterization showed these fines to be crystalline clays with a very small size distribution. These observations suggested that organic material is not required in order to explain MFT properties. Further work focussed on the mineral model for MFT stability and resulted in a closer look at the concepts developed by Yong and Sethi [7]. In broad terms, this model involved particles with large surface areas, and a concomitant high water holding capacity. Not all MFT samples contained significant montmorrillonite or amorphous iron (the major contributors to surface area in the Yong and Sethi mineral stability model). However, the realization that the kaolinite and illite clays are potentially an order of magnitude (or more) smaller than originally thought could more than make up for the absence of montmorrillonite and amorphous iron [16].

In parallel to the ultrafines gelation work by Kotlyar, studies at CANMET were focussing on characterization of floc structures and aggregate morphology to model the bulk behavior of mature fine tailings [3,17–19]. The gellation and floc structure concepts were brought together by describing MFT as a floc hierarchy or by the fractal nature of the flocculated clays. The so called ultrafine fraction of minerals (particles less than 200nm in size) would never settle in the tailings pond to form MFT unless they first formed flocs large enough. These flocs grow and interact to form larger entities in a floc hierarchy, or a structure that can be described by its fractal nature [20].

Direct observation of the MFT from the Clark hot water extraction process revealed a distinct clay structure, very different from that observed in MFT from the OSLO extraction process [17,18]. Figure 4-5 shows the distinct difference between the Clark and OSLO mature fine tailings. There is a more random interaction in the OSLO MFT relative to the predominantly edge to edge orientation of the Clark MFT. Since the OSLO extraction process is less dispersive, the clays are already aggregated and are not as free to orient in the same way as the dispersed Clark tailings clays.

This difference in aggregation is shown in Figure 4-6 where a hydrometer test was used to determine the clay size distribution under natural or dispersive conditions. There is a significant difference in the size distributions determined

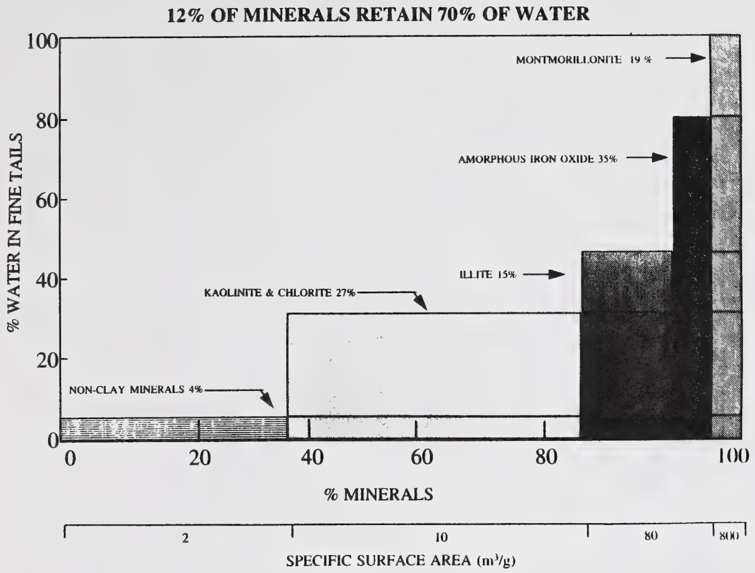


FIGURE 4-3. The mineral control of mature fine tailings properties (from Yong and Sethi [7]).

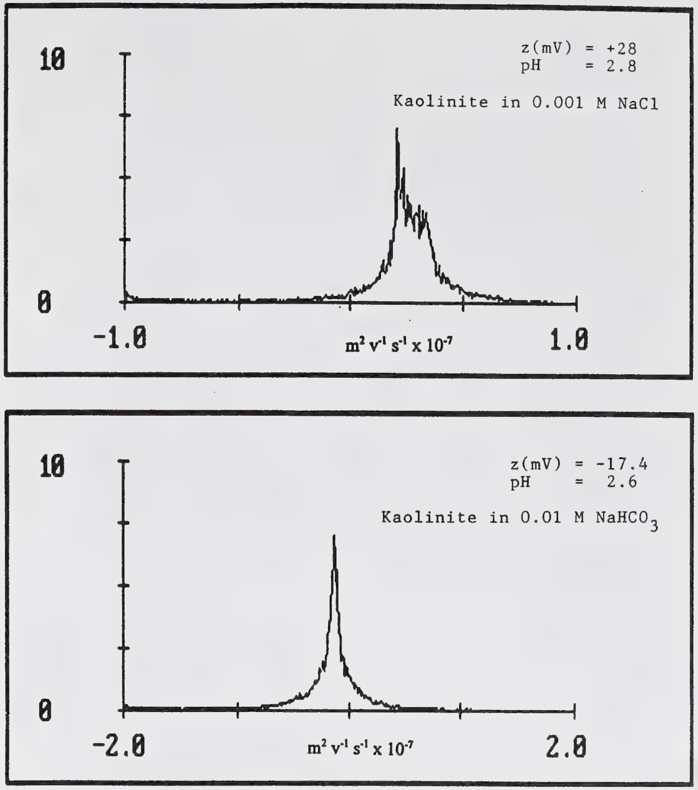


FIGURE 4-4. The electrophoretic mobility (at low pH) of kaolinite in water (top) and in the presence of bicarbonate (bottom). The electronegative character in bicarbonate is similar to that observed for clays in pond water. This was initially interpreted as being due to organic species in the pond water affecting the clay surfaces. The same behavior with bicarbonate and no organics in this model system confirms that a mineral mechanism can account for the clay behavior rather than adsorption of organics from the pond water.

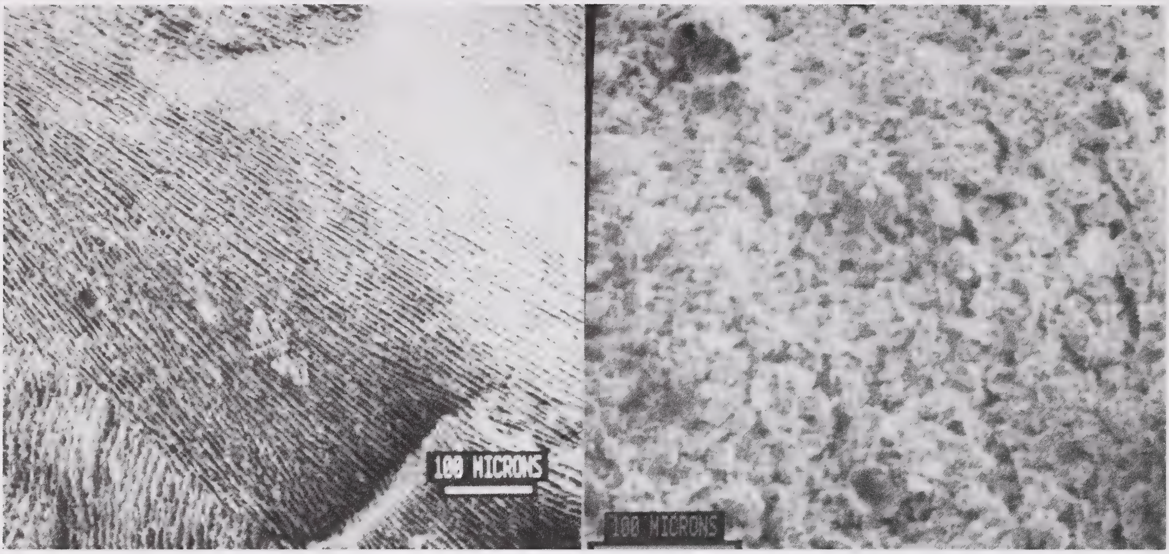


FIGURE 4-5. Scanning electron microscope images of Clark (left) and OSLO (right) MFT showing the distinctly different floc structuring.

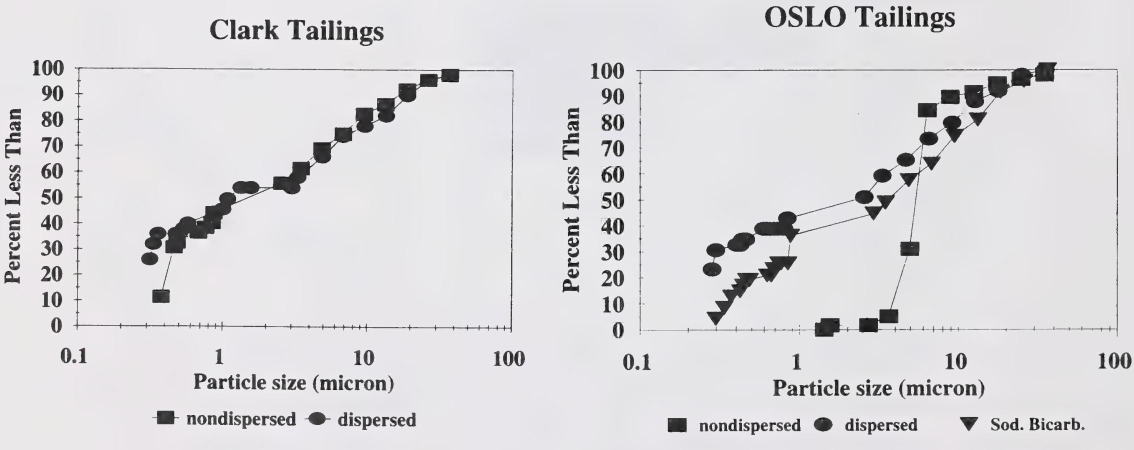


FIGURE 4-6. Hydrometer size distributions determined for dispersed and as received OSLO tailings. The OSLO clays were not completely dispersed except after dispersant was added for the double hydrometer test. The addition of 1000 ppm bicarbonate also has a significant dispersing effect. Similar tests with Clark tailings show no difference between the as received and dispersed tailings, indicating that the Clark tailings clays are already dispersed.

under dispersive and nondispersive conditions for the OSLO MFT proving that the clays are flocculated or aggregated. The dispersing effect of bicarbonate is also shown in this figure. Clark tailings clays are unaffected by the dispersant, indicating that they are already dispersed. These differences in floc structure or how the clays interact determine the bulk properties of the MFT [3].

Figure 4-7 shows the rheological behavior of Clark MFT as a function of sample handling. MFT allowed to sit for 72 hours sets up a structural strength that is manifested by a large yield stress and significant thixotropy (shear thinning behavior, determined from the hysteresis in the shear stress *versus* shear rate curves). The sample which was sheared to break up the structure and then characterized shows a much smaller yield point and lower thixotropy. The sample which was analyzed after introduction to the sample holder, but without shearing shows an intermediate behavior. Figure 4-8 shows direct microscopic evidence of the structural changes with sample handling, analogous to the rheological characterization. The most distinct structuring of the clay components is shown in the sample that was allowed to sit for the longest period of time before observation. The sample which was vibrated and then observed a very short time afterward shows a completely random clay orientation. The sample which was subjected to an intermediate amount of disturbance shows an intermediate amount of structure break up [2,3].

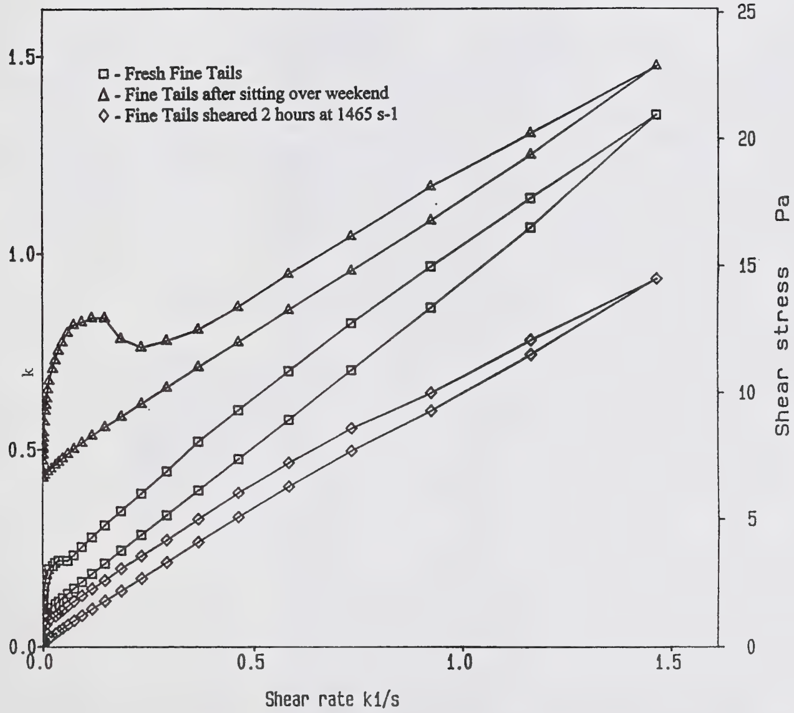


FIGURE 4-7. Rheological behavior of MFT under different conditions. The upper plot of shear stress *versus* shear rate is for an MFT sample that was allowed to sit undisturbed for 72 hours. It has a significant thixotropy and yield stress. The lower plot is for an MFT sample that was sheared to break up the floc structure and then the shear stress *versus* shear rate data collected. This sample shows a significantly lower thixotropy and yield stress. The intermediate behavior is from a sample that was poured into the sample holder and analyzed without first shearing, and without a long time to sit undisturbed.

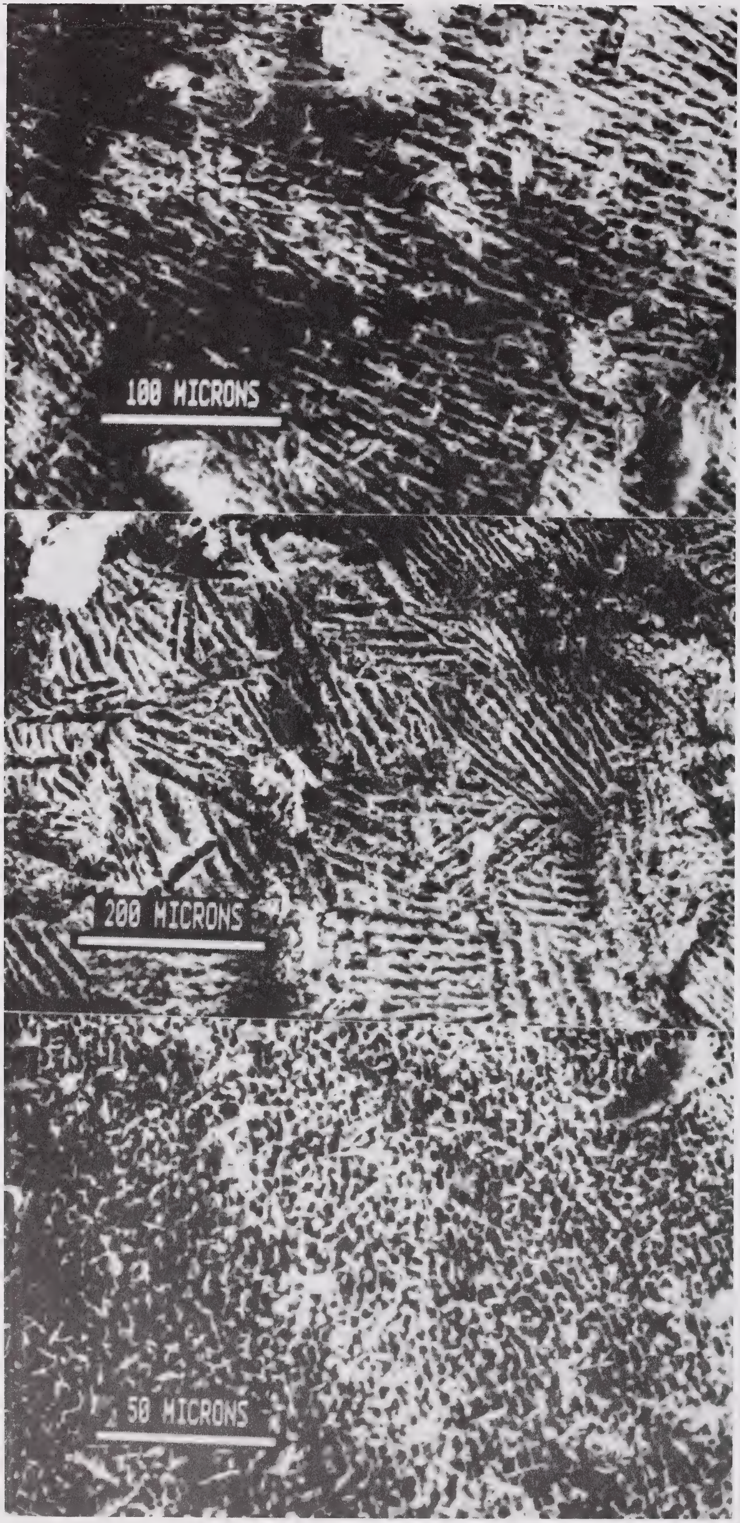


FIGURE 4-8. Microscopic floc morphology for MFT samples allowed to sit for various periods of time. The top photograph is MFT that was undisturbed for the longest period of time. The bottom photograph is for a MFT sample that was vibrated violently and then immediately observed. The middle photograph, which shows an intermediate amount of disturbance in the structure, was vibrated and allowed to sit for a short period of time. These samples were all prepared using cryogenic methods in exactly the same way.

This card house like structure for clays has been discussed by others [6,21,22]. By preventing formation of this intermediate stable structure, or by changing it afterward, a more compact settled material can be formed.

By way of illustration, the clay flocculation and aggregate structures associated with MFT formation can be thought of in terms of the water holding capacity of mineral surfaces. The more mineral surfaces that are exposed, the higher the water holding capacity, and the lower the permeability. If the mineral surfaces are aggregated to each other rather than dispersed, less interaction with the water is possible. At the same time, a less dispersed clay system (OSLO or chemically treated MFT) is stronger due to greater mineral-mineral interactions relative to the dispersed (Clark MFT) suspension where mineral-water interactions dominate. The differences in bulk properties of Clark MFT, OSLO MFT and chemically treated MFT can all be interpreted in terms of this floc structure model. Rheological and centrifugation behavior best illustrate the floc structure concepts shown in Figure 4-9.

Low speed centrifugation studies confirmed that the Clark MFT floc structure has a resistance to settling and that if the particular clay floc structure associated with the Clark MFT is not allowed to form by either physical (centrifugation) or chemical (OSLO process water chemistry) intervention, a lower settled volume can be achieved. A fine tails suspension (approximately 8% by weight) can be centrifuged at 230 g to a relatively compact state. The same suspension which is first

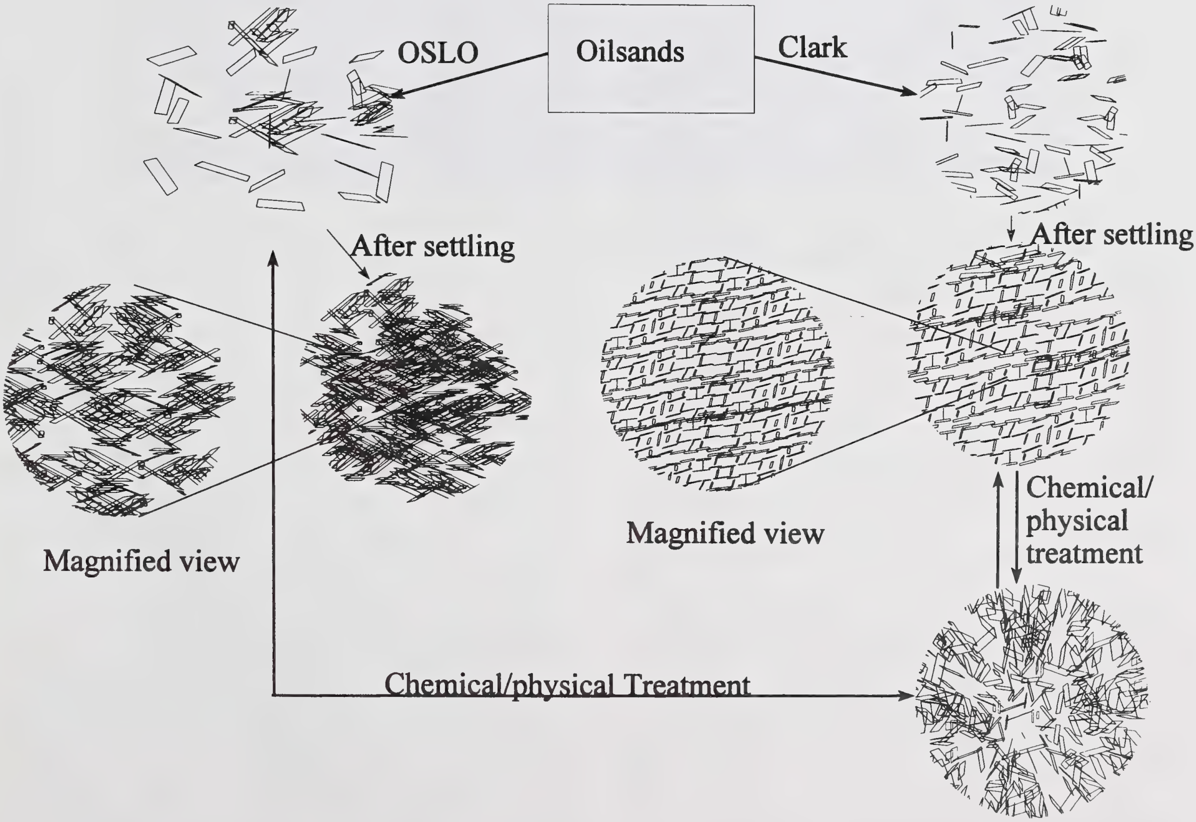


FIGURE 4-9. This illustrates the fractal nature of the MFT floc structure and the potential for chemical or physical manipulation of the structure to produce a material that is more easily consolidated or dewatered.

allowed to settle to a solids content of about 18% (by weight) at 1 g will compact very little at 230 g (Figure 4-10). At 115 g, the fine tails suspension and the settled fine tails compact to the same extent. This indicates that the stable state of MFT can be bypassed if an appropriate force is applied to prevent structure formation. Once the structure is formed, it can be more difficult to collapse or consolidate. In the case of OSLO MFT where solids content is similar but microscopic observation showed a different floc or aggregate structure, both the settled and original fine tailings compact to the same extent at 230 g (Figure 4-11). The rheological characterization of these same Clark and OSLO tailings suspensions showed that the OSLO material shows particle association or structuring at a solids content where the Clark material is still dispersed. This is shown in Figure 4-12 where the elastic modulus (G', the elastic response to a stress that does not break the aggregate structure), of the suspension is measurable for OSLO tailings while the Clark material has no structural character until 18 to 20% solids is reached [23]. Figure 4-13 shows optical images of the floc structural differences in the Clark and OSLO fine tailings where the floc structures are clearly different [19]. These results are discussed in more detail in the appendix.

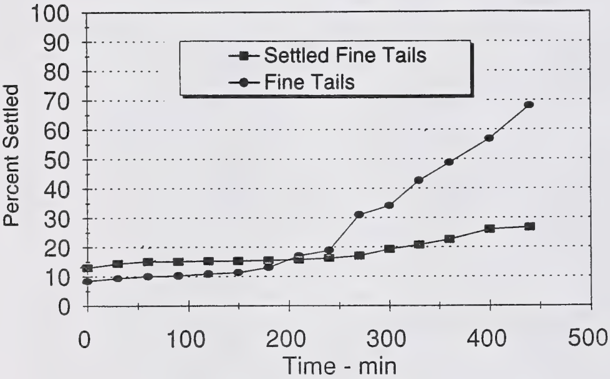


FIGURE 4-10. Centrifugation of Clark tailings and settled tailings. Centrifugation of the settled tailings at 230 g results in very little further dewatering. Centrifugation of the tailings suspension does not allow formation of the stable MFT structure and results in significantly more dewatering.

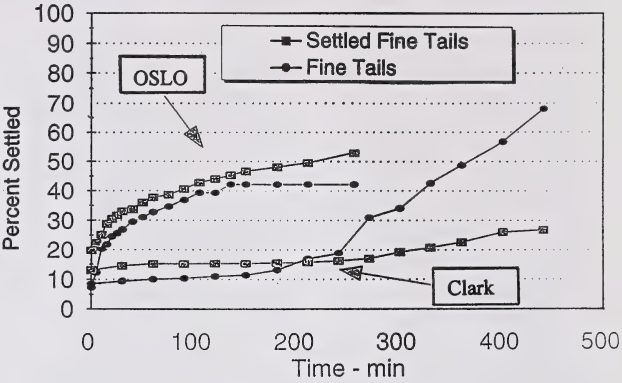


FIGURE 4-11. Centrifugation of OSLO tailings and settled tailings. Centrifugation of the settled tailings at 230 g results in the same dewatering as for the tailings suspension. The OSLO floc structure is not as resistant to further dewatering as the Clark.

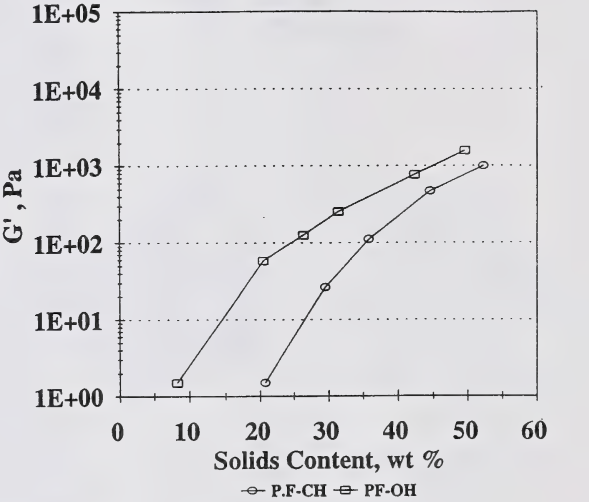


FIGURE 4-12. Rheology of the OSLO and Clark tailings suspensions as a function of solids content. The OSLO material has measurable structural properties (the elastic modulus, G') at 8% solids, whereas the Clark material doesn't exhibit these properties until a solids content of about 20% is reached. This is further evidence of the water chemistry dependence of clay floc structure.

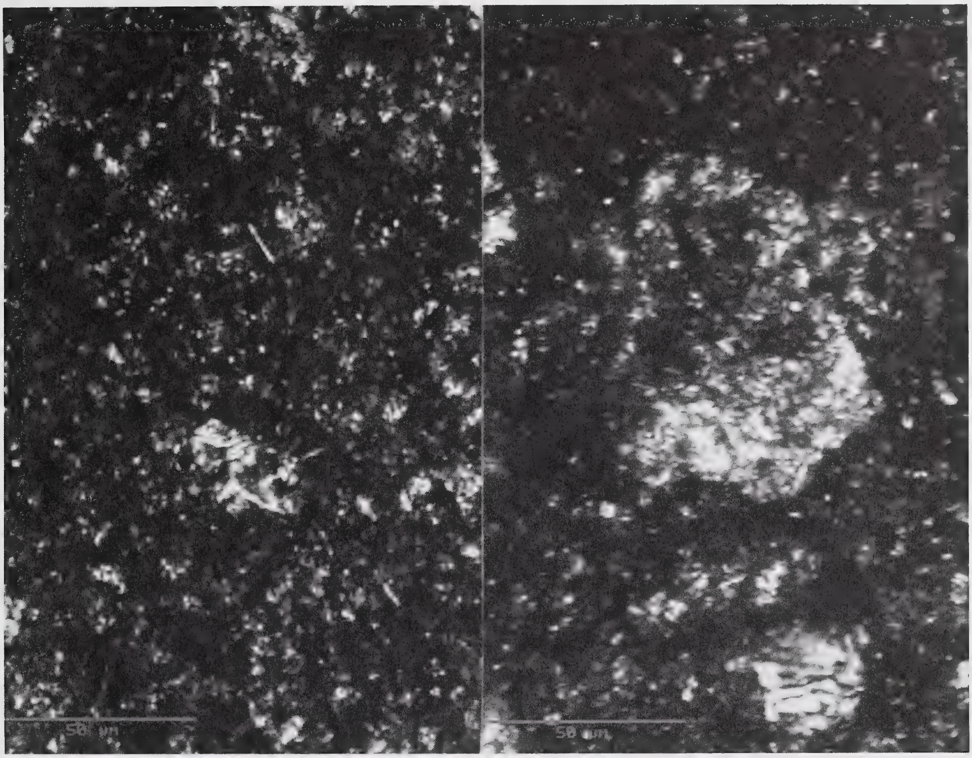


FIGURE 4-13. Optical microscopy of Clark (left) and OSLO (right) tailings. The OSLO tailings have significantly larger aggregates than the Clark tailings. These microstructural differences are responsible for the bulk rheological behavior.

Application of a stress or force to overcome the stable floc structure in MFT is the basis of success of nonsegregating tailings (NST) and freeze thaw (FT) as ways to increase the amount of water released from MFT (increase the degree of consolidation). In the case of NST, a force is applied to the MFT structure by the introduction of sand grains to the clay suspension (which has been chemically treated to produce a stronger structure), resulting in self weight consolidation [24–27]. With FT, the formation of ice crystals forces the clays together, collapsing the floc or aggregate structure, resulting in water release [27–29].

The recognition of floc structure in determining bulk properties resulted in studies to describe MFT behavior from a theoretical viewpoint [21,22,30]. This effort was hampered by the experimental data available and by the limitations of many of the theoretical treatments to suspensions with low solids concentrations. On the other hand, experimental characterization was made difficult by the relatively dilute tailings suspensions which only very slowly develop the 25 to 30% solids content characteristic of mature fine tailings. As a result, permeability and consolidation data are difficult to obtain, although a significant database has been established [4,6,24,28]. Permeability and consolidation are critical properties for the prediction of geotechnical behavior of the MFT deposits.

The difficult and time consuming nature of conventional MFT characterization tests led to the development of a series of index tests by which MFT properties could be quickly compared. Most of these tests probe in some way the floc or aggregate structure of the fine particles that make up the MFT. These index tests illustrate that this structure (and the bulk properties) are dependent upon the water chemistry of the suspension, particularly the pH. The weakest floc structure is at approximately pH 8.4, the pH defined by the bicarbonate buffer. Figure 4-14 shows the pH dependence for the electrosonic amplitude (ESA) which measures the electrostatic interaction of the clays in suspension. A larger absolute value means a larger electrical repulsion between particles. The G' also shows the most disperse system at pH 8 to 9. Figures 4-15 and 4-16 show similar pH dependence for the water released with low speed centrifugation and the specific resistance to filtration.

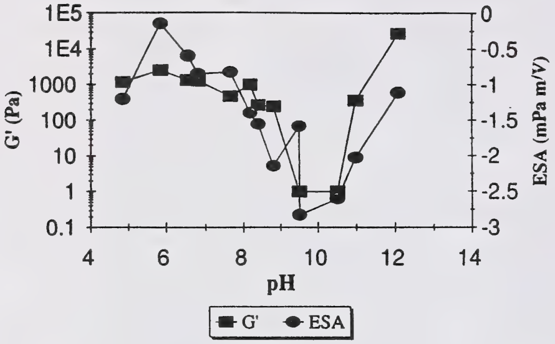


FIGURE 4-14. Electrosonic amplitude (ESA) and elastic modulus (G') as a function of pH. The most weakly flocculated clays correspond to a high absolute value of ESA and a low G'. This occurs at a pH of between 8 and 9 corresponding to the pH determined by the bicarbonate buffer.

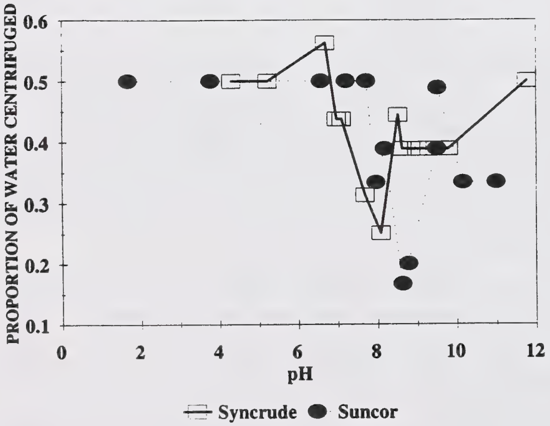


FIGURE 4-15. Water release during low speed centrifugation from MFT as a function of pH. The minimum in water release corresponds to the bicarbonate buffer pH.

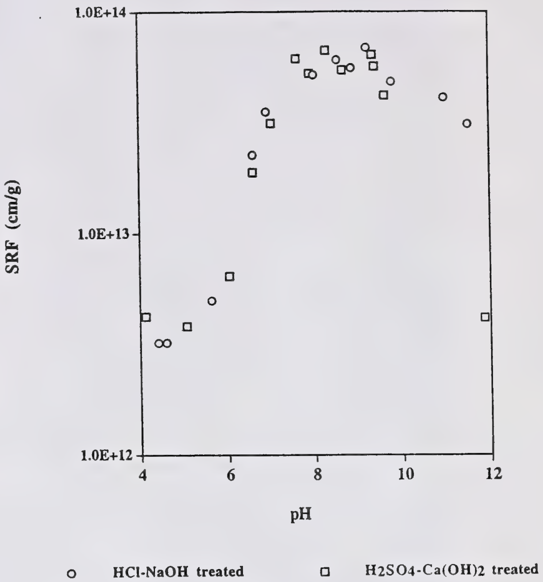


FIGURE 4-16. Specific resistance to filtration as a function of pH. The most resistance to filtration occurs at a pH of 8 to 9, corresponding to the pH determined by the bicarbonate buffer.

All of these properties indicate a more weakly interacting clay suspension at a pH between 8 and 9. Since this pH corresponds to the bicarbonate buffer pH, controlling pH controls bicarbonate concentration and MFT properties.

4.2 Summary

Mature fine tailings are a weakly flocculated system of fine clays with a floc or aggregate structure that is incapable of supporting larger particles that could stress the floc structure in order to initiate consolidation. The water holding capacity of the MFT is largely controlled by the mineral composition and the floc or aggregate mineral structure can be manipulated chemically (by changing the water chemistry of the suspension fluid) or physically (by centrifugation or by freeze thaw strain) to enhance dewatering. Changing the MFT floc structure is the underlying mechanism behind the creation of nonsegregating tailings, freeze thaw, and other MFT treatment processes. The water chemistry and pH dependence of MFT properties, and the recognition that these properties are a function of the floc or aggregate structuring of the MFT clays is important in predicting the long term behavior of tailings deposits.

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4.4 APPENDIX — Index Tests for the Characterization of MFT

4.4.1 Introduction

This appendix provides descriptions of several index tests developed to permit rapid comparisons of various MFT samples with regard to their floc or aggregate structure. These tests, although empirical, provide important comparative information about the samples. The tests include quantification of rheological and electrokinetic properties, measurements of specific resistance to filtration, accelerated settling tests, the double hydrometer test (aggregation index), and direct observation using optical and scanning electron microscopy. These index tests determine the extent to which MFT aggregate structure is influenced by the caustic environment found in Clark hot water tailings. In order to investigate MFT properties as a function of pH, the pH was adjusted in the acid region by adding either HCl or H_2SO_4 and, in the basic region, by adding either $Ca(OH)_2$ or NaOH.

The elastic modulus, G', (which is related to the degree of dispersion), the amount of water released during low-speed centrifugation, the specific resistance to filtration, and the electrophoretic mobility all showed strong pH dependence. The least desirable MFT properties were noted at pH about 8.5, corresponding to the pH where bicarbonate concentration is at a maximum.

The microscopic studies along with the microelectrophoresis show that bicarbonate concentration is directly related to fundamental clay properties such as electrophoretic mobility and floc morphology. The chemistry of the fine tailings system is complex and there are clear relationships between several constituents that are likely important. For instance, although added calcium (as chloride or sulphate) increased the degree of flocculation, the effect was not as pronounced in the presence of excess bicarbonate.

It is possible to convert Clark hot water type MFT to OSLO type MFT and *vice versa* by manipulating the bicarbonate chemistry. This was clearly illustrated in the microelectrophoresis where fine tailings from the Clark and OSLO processes were compared in their respective pore waters, as well as in ideal bicarbonate solutions. In fact, all of the index tests show a clear link between MFT properties and pH, corresponding to the pH dependence of bicarbonate ion concentration. Although bicarbonate concentration was correlated with electrokinetic behavior and can be identified as the most important index, the experimental results also point to other factors and do not rule out other mechanisms (including the possible role of organics) that happen to be correlated with the bicarbonate pH dependence. Further work is required to understand the individual roles of bicarbonate concentration and pH in determining the floc structure of MFT and the subsequent bulk properties.

The equilibrium of aqueous CO_2 species with pH is well known and is shown in Figure 4.4-1. Figure 4.4-2 shows the experimentally determined total carbonate concentration in mature fine tailings 7 days after pH adjustment revealing a pH dependence similar to that of the ideal bicarbonate system. This is not surprising since the major anion in MFT is bicarbonate. The index tests also show behavior which correlates to this bicarbonate concentration.

Rheological parameters measured at pH <7 or >10 indicate a highly flocculated fine tailings system while, at intermediate pH values, the MFT fines are considerably more dispersed. Plots of G' (elastic modulus) *versus* pH for both Syncrude and Suncor fine tails pH-adjusted with H_2SO_4 or $Ca(OH)_2$ produced similar trends in behavior. Values of G' are at their lowest in the pH range 8 to 10. These low values of G' are indicative of a dispersed fine tails system. With higher or lower pH values, G' increases, indicating that the degree of flocculation has increased. Electrokinetic sonic amplitude (ESA) (Pa/mV) data are related to the degree of charge

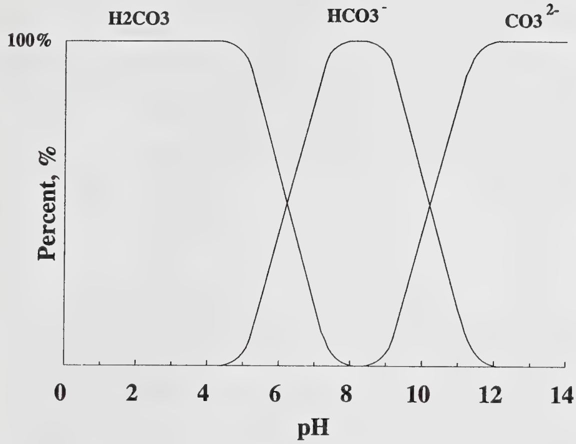

FIGURE 4.4-1. Equilibrium of aqueous CO_2 species with pH (from Bohn, H.L., McNeal, B.L., and O'Conner, G.A., "Soil Chemistry," 2nd Edition, p 96, Figure 3-4A, Wiley-Interscience publication, John Wiley and Sons, 1985).

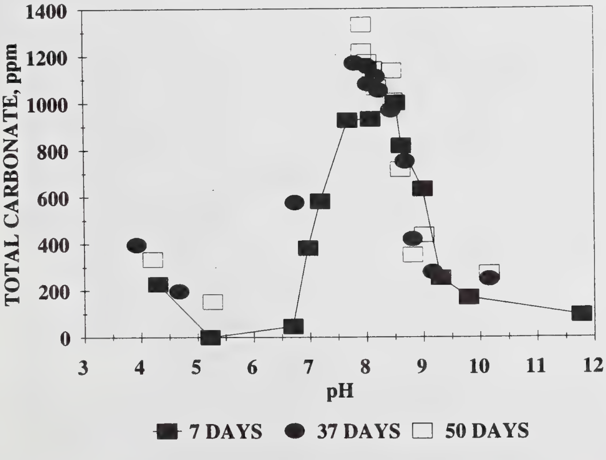

FIGURE 4.4-2. Syncrude MFT total carbonate *versus* pH.

imparted to the particle surfaces as well as the relative sizes of the clusters and the degree of flocculation (or dispersion). A greater degree of flocculation is indicated by smaller negative ESA values in the systems studied. The signal also decreases when there is an increase in inertia due to the size of the flocs. The correlation of ESA with G' determines whether or not a system has a tendency to flocculate and, if so, to what extent and how strongly. As discussed earlier, this does not imply the exact mechanisms by which this behavior occurs.

Figure 4.4-3 shows the electrophoretic mobility (EM) of the Suncor pond 1 mature fine tails diluted in their associated water as pH is decreased by the addition of dilute HCl. The specific conductivities are traced at the same time as the pH. This graph indicates that the net negative electrical charges at the particle/fluid interface are decreased as pH in the continuous phase is reduced. It also shows that the H^+ ions are potential determining which means reduced zeta potential with pH reduction. This indicates that the samples are most negatively charged at basic pH and therefore more dispersed.

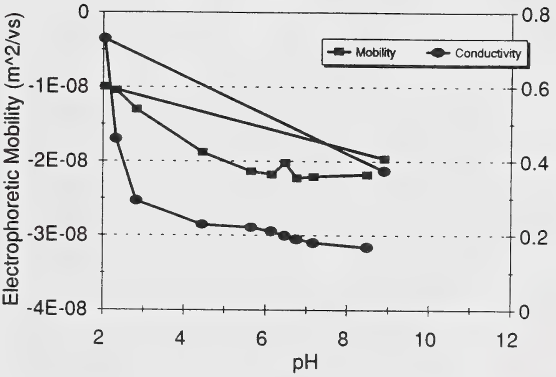

FIGURE 4.4-3. EM and specific conductivity *versus* pH for Suncor pond 1 fine tails in associated water to pH 2.0; ( ) shows addition of NaOH after pH 2.0.

The specific resistance to filtration (SRF) is related to the permeability of MFT and is an indicator of the floc structure or strength and degree of particle dispersion. The more dispersed the MFT, the denser the final compacted sediment and the lower the permeability. In addition, a more dispersed MFT is more likely to blind the filter and greatly increase the measured SRF. There is a pH dependence of the SRF that corresponds to the pH dependence of the bicarbonate concentration.

As shown earlier, water release during centrifugation is minimized when the MFT solids are dispersed and the results show that the amount of water released has a pH dependence corresponding approximately to the pH dependence of bicarbonate ion concentration. At higher (>10) or lower (<7) pH, the MFT solids are flocculated or aggregated in a way that results in a greater proportion of water being released during low-speed centrifugation.

The differences in the degree of aggregation were also confirmed using the double hydrometer test which compared aggregate size in dispersed and non-dispersed suspensions. These results were shown earlier for the Clark and OSLO MFT. The Clark MFT clays were significantly more dispersed than the OSLO clays.

The morphologies of Clark and OSLO mature fine tailings have been shown to be completely different when observed using cryogenic scanning electron microscopy on fast-frozen samples, as well as with optical microscopic methods on unfrozen samples. Table 4.4-1 outlines the observed morphologies along with some of the bulk properties that are indicative of a conversion of OSLO to Clark and Clark to OSLO mature fine tailings by the manipulation of the bicarbonate chemistry. This correlation is not necessarily indicative of a mechanistic role for bicarbonate since it has been noted that bicarbonate concentration and pH also affect bitumen morphology in MFT. The role of other species (inorganic or organic) in controlling MFT morphology and therefore the bulk properties cannot be ruled out.

The results of all of the index tests show a pH dependence of the microscopic and bulk MFT properties. The Clark MFT are relatively dispersed at pH in the range of 8 to 10, corresponding to the maximum bicarbonate concentration. Bicarbonate is the most abundant anion present in Clark MFT due to bicarbonate in the oil sands as well as the basic (caustic) tailings water which, over time, has equilibrated with CO_2 from the air.

Comparison of OSLO and Clark MFT showed that the OSLO MFT are less dispersed. By altering the pore water chemistry it is possible to convert Clark MFT to less dispersed MFT that exhibit properties similar to those of the OSLO MFT. Conversely, by adding bicarbonate it is possible to convert OSLO MFT to MFT having properties (both microscopic and bulk) resembling those of the more dispersed Clark MFT. Clark MFT that are decarbonated or brought to acid pH (to allow closer interparticle association, more flocculation) and subsequently shifted to neutral pH, have properties similar to the OSLO MFT.

TABLE 4.4-1. Microscopic floc structure, rheology, and water release for various MFT treatments. The mean value is the average microscopic (optical) reflectance of the sample which is indicative of the aggregation of the sample. The Clark MFT are the least aggregated of the samples investigated. The structure observed in the scanning electron microscope (SEM) is a subjective assessment of the sample morphology. The elastic modulus (G') is also a measure of the degree of aggregation. The lower the G', the greater the dispersion of the sample. The specific resistance to filtration (SRF) is another empirical test that is related to the floc or aggregate structure. The higher the SRF, the smaller the flocs or aggregates and the more resistance the sample has to filtration.

Date	Sample identification							
	Clark MFT	OSLO MFT	OSLO MFT + 1500 ppm bicarbonate	HCl to pH 6.0	H_2SO_4 to pH 6.0	HCl to pH 6.0 then $Ca(OH)_2$ to pH 8.0	H_2SO_4 to pH 6.0 then $Ca(OH)_2$ to pH 8.0	$CaSO_4{\cdot}2H_2O$ 800 ppm Ca
Mean value of reflectance	2.94	2.00	1.93	2.18	2.63	2.26	2.18	1.87
SEM structure	yes	none	yes	very little	very little	none	none	very little
G'	44	359	179	415	360	402	454	720
%Water release (after one month)	0	7	8	10	10	10		
SRF × 10^{-13} cm/g	6.84	1.2	1.3	1.65	2.63	1.44	.862	.505

4.5 Part 1: Rheology

4.5.1 Objective

Rheological measurements were tools used to study the varied viscoelastic properties of mature fine tailings and their components. The tailings flow properties which were inherent or modified (by handling over time, by shearing history, dilution, drying, modified by chemical treatments, produced from various extraction processes or modified by flow enhancing agents) were all investigated with the goal of understanding fine tails behavior and interparticle interactions.

4.5.2 Methodology

Oscillatory and steady state and relaxation rheological measurements were conducted on a Bohlin VOR rheometer using a concentric cylinder sensor system (model C25) which has a viscosity range of 10^{-4} to 10^{5} Pa·s. The C25 has a large sensing area and is the most sensitive of the standard concentric cylinder types available. The gap spacing is 2.5 mm and the radius of the rotating cup is 27.5 mm. A sample volume of 13 mL is required to cover the stationary bob which is 375 mm in height. Torsion bars of 1, 11, and 90 g·cm were used, depending on the volume fraction of pond tailings studied. The system was operated at 20.0 ± 0.5°C. A Neslab RTE 110 circulating bath was used to maintain stable temperatures.

Steady-state viscometry (shear stress *versus* shear rate) curves were obtained in 55 steps over a shear rate range of 5.8×10^{-3} s^{-1} to 1.46×10^{3} s^{-1}. Up/down curves were obtained to measure the degree of structural breakdown due to shearing.

In dynamic testing, a measure of the linear viscoelastic properties of the MFT was obtained by determining the linear viscoelastic region using strain sweeps. Strain sweeps were performed at 1 Hz. Oscillatory sweeps in the linear viscoelastic region were performed at a strain of 0.008 in 12 steps ranging from 0.009 Hz to 1.5 Hz.

Relaxation studies after two steady-state viscometry sweeps were performed at a nominal shear strain of 0.1787 and a strain rise time of 10 s over a 15-minute period.

Pulse shearometry is used for low strain low deformation measurements of viscoelastic systems in near static conditions. The shear modulus is measured using a Rank Mark II pulse shearometer, originally designed for studying the strength of viscoelastic systems of the food and drug industry (gels, pastes, and yoghurt). In this technique the sample resides between two parallel plate stainless steel discs, each of diameter 2.5 cm, contained in a glass cylinder such that their separation can be varied with a screw cap and measured by a dial gauge. Each disc is connected to a piezoelectric crystal (lithium chloride, similar to the types used in expensive audio cartridges) with a thin perspex shaft. A small displacement of the lower crystal is produced by a pulse generator (1×10^{-4} rad) allowing the outer edge of the disc to rotate by about 1 micron in its own plane. This pulse is maintained for 21 ms. The motion of the lower disc transmits a shear wave through the viscoelastic sample to the upper disc, which then turns. The signal is converted to an electrical signal monitored by an oscilloscope. The initiation of the pulse is recorded by the receiver crystal as the beginning of a linear portion of the oscilloscope trace, and the arrival of the pulse at the upper disc is indicated by a damped sine wave. The time span from the initial triggering of the lower disc to the first peak of the first wave recorded on the oscilloscope is used to calculate the propagation velocity V. The gap width is varied from 3 mm to 25 mm. The frequency of the shear wave is 200 Hz. The strain is extremely low and therefore it is expected that the system will exhibit linear viscoelastic properties. Shear modulus is calculated from a product of the density of the material and the square of the velocity of propagation.

4.5.3 Results

Viscoelastic properties exhibited by oil sand fine tails (MFT) in steady-state and dynamic viscometry indicate the presence of aggregates [1]. Changes in the time scales of fine tails can be used to evaluate the effect of physical or chemical treatments which cause changes in MFT structure [2]. These changes are observed in values of yield point obtained in steady-state viscometry and G' (elastic modulus) obtained in dynamic viscometry. Other time scale measurements were traced using pulse shearometry, where the effects of ultracentrifugal stress (samples were centrifuged in a Beckman L8-80 ultracentrifuge at 17000 rpm then redispersed). were studied by tracing restructuring of the elastic flocs with time. When the particles were allowed to restructure with time, the shear modulus followed a first order reaction kinetic curve [5].

Rheological properties of MFT correlate well with pH showing highly flocculated fine tails at extreme ends of the pH range [3]. Minimum values for elastic modulus and yield point for Syncrude at pH 9 and for Suncor at pH 9.5–10.5 indicate dispersed fine tails. On either side of this minimum the degree of flocculation tends to increase.

In the determination of differences in the fine tails produced in different processes, the sediments shear modulus was measured and found to differ with the OHWE process forming more elastic particulate structures than the CHWE

[6]. This is consistent with the the findings of the oscillatory and steady state measurements obtained for OHWE than for CHWE fine tailings [4]. Differences were greater at lower solids contents and became smaller as solids content increased. The absence of elastic modulus and yield point values for Syncrude CHWE fine tailings having less than 26 wt% solids and for Suncor CHWE fine tailings at 21 wt% solids indicates highly disperse systems. For OHWE fine tails, elastic modulus and yield point values were measured at 8 wt% solids for both Syncrude treated tails and Suncor tails, indicating the presence of aggregates.

All these techniques were also used to study the effects of dilution of MFT where a critical volume fraction for flow was identified as the storage (elastic, shear) and loss modulus as well as the yield stress followed a power law relationship with volume fraction. The power n was consistent for each property and with a flocculated system [7]. In addition these techniques were found appropriate for determining the degree of modification of the elastic floc structures required to enhance flow when treated by chemical flow enhancers. This showed that MFT can be modified to reorganize the aggregates and enhance flow [8].

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4.6 Part 2: Electrokinetics and Surface Properties in Mature Fine Tailings Stability and Structure

4.6.1 Objective

Electrical repulsive forces are the primary forces in simple electrostatically controlled disperse systems, where particles with colloidal properties have highly charged surfaces. The nature of the particles forming MFT in the tailings ponds is determined by the processing history of the ores and the nature of the continuous media. The particulate surface properties, sizes, and shapes thus dictate the final interparticle interactions and hence the structures in concentrated systems.

The question of the dominance of the total repulsive forces over the attractive forces could not be answered without detailed experimental investigation of the contributions of each of the component forces. The net repulsive forces arise from several major vector component forces such as electrical (V_E), steric (V_S), and capillary/hydration (V_C/V_H). The attractive forces arise from van der Waals (V_V), (particles nature and distance of approach) and hydrophobic (water repelling organic molecules bound to surfaces of clays, mineral oxides, and oil droplets) or oppositely charged electrostatic surfaces. The net interactive force V_T is governed by a summation of individual component vector forces:

$$V_T = V_E + V_V + V_S + V_C + V_H$$

Extensive research was focussed on (1) understanding the nature of the particles surfaces that interact in MFT, *i.e.*, the role of the surface functional groups on the clays, mineral oxides, and bitumen, whether these were acquired naturally or caused by adsorption; or (2) whether the surface properties and behavior were mainly because of the composition of the continuous medium; (3) whether the final behavior and arrangement in a given space were due to the types of particles interacting in the given space. To answer some of these questions, considerable research was done to investigate the surface chemistry, electrokinetics, and flow properties of components derived from MFT, whole MFT from different sources, and fine tailings derived from different processes.

CANMET investigated the components of the heterogeneous population of clay particles that contribute to the hydrophobicity of the particle surfaces. Steric factors and the electrical contributions of the component particles and their modified surfaces and populations were also studied. In collaboration with Illinois Institute of Technology and Columbia University, which investigated the role of capillary forces and the contributions to steric forces, respectively, an understanding of the energy network for MFT stability was defined. The surface and electrical properties, together with the rheological flow properties, provided a clearer understanding of the particulate organization in a given space under low and high stress as the systems moved from dilute to concentrated. This was the premise by which the fundamental colloidal studies of MFT stability and the particles organization in space were undertaken in the past five years. The effects of components making up the MFT, their influence on each other in space, and their stability and flow characteristics were investigated in comprehensive studies. The following summarizes some of the major parts of this work.

4.6.2 Methodology

Whole fine tails were initially obtained from the Suncor plant # 3 tailings pond. These MFT samples were subjected to CANMET laboratory separation techniques designed to liberate the component clays and oil from the associated water for the study of individual components. The component clays derived from clays were extensively characterized for surface and electrical properties in various media and pH conditions, and later modified chemically to study the effects of component functional groups on flow properties and stability. These clays were compared with model kaolinites, illites, and silica in a common electrolyte medium or pond water. They were also compared with the model clays using specifically adsorbed organic components such as humics and asphaltenes derived from oil sands sources.

For, further verification, subsequent comparisons were made with MFT from Suncor pond 1 and pond 2 of June '91, pond 1 of June '93, pond 1 of Oct '94, and Syncrude June '91. Other comparisons were made with an OSLO MFT sample obtained in June '91. Finally, comparisons were made on the tailings samples derived from a pilot study designed to compare two ores processed by Clark and OSLO extraction processes using various waters.

Three clay fractions denoted B = bottom layer, M = middle layer, and T = top layer clays were separated using high-speed ultracentrifugation in their natural waters (Beckman L8-80), microtome slicing, and freeze drying. The S = supernatant water was saved for reconstitution.

The electrical properties (electrophoretic mobility distributions) of the dilute suspensions were determined with a PENKEM System 3000 automatic microelectrophoresis apparatus. For the concentrated suspensions, the Matec MBS 8000 and an automatic titration system designed by CANMET were used, not only for charge density studies, but also for noting the pH effects in various media.

4.6.3 Results

4.6.3.1 *Clay Components from Mature Fine Tailings and Model Systems*

Investigations were geared towards full characterization of the surface properties of these clays [1] and comparing their behavior with model systems such as cation-exchanged clays with adsorbed humics and asphaltenes derived from the oil sands bitumen [2]. The effects of the organics adsorbed on the clays, whether obtained naturally through the process [1] or adsorbed from the water [3,5], were shown to change the electrophoretic mobility distributions of the clays separated from the MFT, model kaolinites, illites, and designed clay-organic complexes. The role of the soluble organics from the water in modifying the surface properties was confirmed by Columbia University in the study of adsorption of pond water on kaolinites and illites [6]. These organics were selective to the types of clays as was shown previously in the model systems [2,6] which resulted in shifts in the isoelectric points. Some of these initial investigations were summarized in an overview of the surface properties, reactivity, electrokinetics, and interactions of the MFT clay fractions [4]. This study showed that the electrokinetic properties of clay particles are affected by the nature of the water in which they are suspended, in this case the pond water and the components making up the pond water [4,5,7]. Thus the roles of the inorganic carbonates (dissolved in the water as bicarbonate/carbonate/hydroxide buffer) in relation to the roles of the dissolved organics and the adsorbed carbonates were thoroughly investigated. By titrimetry, the organics were differentiated relative to the bicarbonates [5] and it was shown that both species impart negative charges to the particles and contribute to polydispersity. However, one specie was preferential to the edges of the clay particles. Carbonate particles adsorbed onto the surfaces were more destabilizing than the free ions, which were stabilizing. The model clays without the carbonate particles appeared to be more dispersed in the pore waters and in a synthetic buffer [8]. The clays followed behaviors close to classical DLVO type previously shown for a natural heterogeneous coal associated mixture of kaolinites, montmorrillonites, and quartz [9].

4.6.3.2 *Real Mature Fine Tailings Samples*

Having found that the clays making up the MFT displayed unique properties which were consistent with the sand, carbonate, and organics contents of the MFT, the whole MFT samples were studied for comparison on the basis of inorganics, organics, and processing history in their natural waters. The basic comparisons of the physicochemical and surface properties of typical Clark and OSLO fine tailings [8] showed that the latter exhibited less stability. Further investigations were conducted using dilute dispersions of these MFT samples to elucidate the roles of calcium bicarbonate/carbonate species in electrokinetic behavior.

The mechanisms behind the electrokinetic behavior (dispersion) of the four fine tails in their associated waters were investigated as a function of pH, which was modified by HCl/NaOH at controlled additions per unit area of particles. The basic differences between the Clark and the OSLO fine tails were studied by modifying the continuous phases until the OSLO and Clark fine tails behaved similarly. The reasons behind the differences were explored using further changes to the ionic content of the continuous media. Another study focussed on the behavior of one Clark MFT sample as concentrations of calcium compounds were varied in the continuous medium. Electrophoretic mobility distribution (EMD) spectra were used to study adsorption mechanisms and charging mechanisms as a function of pH as the system responded to monoprotic (1:1) and diprotic (2:2) acids and bases, and calcium species. The role of calcite in electrokinetic (electrostatic component of DLVO) stability was determined. The latter was a continuation of earlier studies on the roles of bicarbonate/carbonate ions in MFT stability [5,7,10,11].

Electrophoretic Mobility Distributions (EMD) and Bicarbonate/Carbonate and Calcium Species with pH. In this work, systems of four fine tails were compared to understand why the OSLO and Clark fine tails differed. The large differences in behavior were attributed to differences in the surface properties of the particles, which is a product of the processing history and the composition of the water-continuous phase. Changing the continuous phase to bicarbonates only while varying the concentrations of $CaCl_2$ showed that the four MFT samples displayed the same trends for pH decrease and return back to basic, but the magnitude of the charge imparted to the systems differed. OSLO MFT appeared to absorb smaller quantities of Ca^{2+} and H^+ ions than the three Clark MFT. Ca^{2+} caused charge reversal only when it slimed onto the particles as a cation complex and formed a coating. The carbonate ions were specifically adsorbing and imparted negative charges to the particles, causing increased dispersion of the particles. Combinations of adsorbed ions changed the surface properties of the particles, depending on pH, reagents, and the sequence of reagent additions to the suspensions.

The various calcium forms destabilized by different mechanisms, depending on whether they were solids in the presence of potential-determining ions or precipitated hydroxides, which were either neutral or positively charged, or occurred as complexes. The mechanisms of charging of fine tails were detailed and the results were used to explain the causes of destabilization or flocculation of particles in a concentrated suspension. The exact role of calcite in stabilization/destabilization was determined using the electrokinetic data.

Electrophoretic Mobility Versus *pH.* Electrophoretic mobility of Suncor pond 1 MFT diluted in their associated waters as pH was lowered with the additions of dilute HCl was investigated. The specific conductivities are traced concurrently with pH. The electrical charge at the particle/fluid interface decreases as pH is reduced in the continuous phase. The H^+ are potential-determining ions which means reduced zeta potential with pH reduction. These data confirm that the samples are most negatively charged at basic pH and, therefore, greater dispersion was expected. If HCO_3^- ions were the most dominant charge-determining ions by specific adsorption, then reduced mobilities in this work would also be a function of the relative amounts of HCO_3^- ions present on the surface and in the continuous medium. The return to basic pH was followed using EM and conductivity data. For this system there was little hysteresis in EM, suggesting that the charge-determining species are reversible for this system at basic pH. The results also suggested that the bicarbonate was not wholly responsible for the degree of dispersion between the particles. This work was a continuation of previous studies on the roles of the bicarbonate/carbonate ions in determining the electrical charge and hence the electrical repulsion between MFT sample particles [1–5,7,10,12]. In this detailed study, all MFT, including OSLO, behave similarly in excess $NaHCO_3$-continuous media as pH is reduced [10].

Electrokinetic Sonic Amplitude (ESA) and Elasticity. The ESA (Pa/mV) data are good reflections of the degree of charge imparted to particle surfaces as well as the relative sizes of the clusters in terms of whether the system is more flocculated (less dispersed), which is indicated by lower negative ESA values in the systems studied. The signal decreases when there is also an increase in inertia, which is mainly due to the size of the flocs. The correlation of ESA with the elastic modulus determines whether or not a system has a tendency to flocculate, and if so, to what extent and how strongly it is flocculated. We have used this correlation in our work for examining the pH-dependent behavior of the Clark MFT. This does not by any means indicate the exact mechanisms by which this behavior occurs, since these are all bulk responses to given changes in the water-continuous media. Charging mechanisms and adsorption behavior are elaborated in another study using microelectrophoresis [10].

One high-caustic Clark (Syncrude) MFT in concentrated form (as received) was studied in order to emphasize the relationships between viscoelastic properties and electrokinetic sonic amplitude, which is a measure of the degree of repulsion (dispersion) or attraction (flocculation) between particles in a concentrated suspension. The samples were studied as a function of pH, using $H_2SO_4/Ca(OH)_2$ and for comparison with another Clark fine MFT, HCl/NaOH in concentrated form were used as pH modifiers. Behavior was also followed as a function of time [12].

ESA and elastic modulus (G') as a function of pH for a typical Clark (Suncor pond 1) MFT showed correlations of high degrees of dispersion (high negative ESA) with low elastic modulus of the concentrated slurries in the basic pH (8.5–9.0). At low pH the particles are more flocculated and ESA is decreased (less negative), while elastic modulus becomes high. The converse is also true. Analogous plots were made of ESA and total bicarbonate measured in the continuous phase at each pH. The high degree of dispersion (highly negative ESA) is consistent with high bicarbonate content found in the associated water at basic pH. This observation is consistent with the role of bicarbonates as a dispersant for most colloidal systems [5–8], and is in agreement with other rheological, filtration, and permeability data [11].

4.6.3.3 *Interparticle Interactions, Electrokinetic and Surface Properties*

The interparticle interactions of fresh tailings processed by the OSLO and Clark processes were investigated in correlations of the surface properties, electrokinetics, and flow properties [13]. The electrokinetic and rheological behavior of these materials was consistent with previous conclusions on the effects of the continuous media, and the effects of organics on the behavior of the MFT. Prior to this study extensive correlations were made with flow properties of concentrated clays and MFT which demonstrated that the MFT particles were organized into a floc hierarchy depending on surface properties, electrical properties, and handleability [4,12,14–19]. Others studying capillary pressure forces have demonstrated that the organization of the coarse and fine particles was directly related to the magnitude of the repulsive capillary forces, where the forces between the fine particles were greater than those between the coarse particles. This is consistent with observations on the electrokinetic and flow properties observed for the same systems [20].

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4.7 Part 3: Specific Resistance to Filtration

4.7.1 Objective

The specific resistance to filtration (SRF) or filterability of a suspension is a quantitative measure of its dewaterability, which is related to the permeability.

SRF is obtained from a pressure filtration experiment carried out under standard conditions [1]. In this filtration process the water must permeate the suspension solids before draining out. The more permeable the filter cake, the smaller the filtration resistance. Therefore, the SRF is related to the permeability of the suspension. Although direct measurement of FT permeability is possible, it is far more complicated and time consuming.

SRF is based on a theoretical model of the filtration. According to Darcy's law and assuming that (1) the filter cake is incompressible and (2) the resistance of the filter media (filter paper here) is negligible compared to that of cake, the following equation can be used to describe constant pressure filtration behavior:

$$(1) \quad \frac{1}{A} \cdot \frac{dV}{dt} = \frac{A\Delta P}{\mu r c V}$$

In the above equation

V = volume of filtrate (cm^3) at time t

A = filter area (cm^2)

ΔP = applied pressure differential (kPa)

μ = filtrate viscosity (Pa·s)

c = solids content of MFT (g/cm^3) which should be determined separately.

r = specific resistance of cake to filtration, *i.e.*, SRF (cm/g).

Integration of Eq. (1) for a batch filtration leads to a mathematical expression for specific resistance to filtration

$$(2) \quad SRF = \frac{2\Delta P A^2 b}{c\mu}$$

where b is the slope of the graph of time/volume against volume in a unit of s/cm^6.

4.7.2 Methodology

Specific resistance to filtration (SRF) is measured with a pressure filtration setup (supplied by Micro Filtration Systems). It consists of a stainless steel cylinder with 200 cm^3 capacity and a control panel, and can apply pressures of up to 700 kPa to the MFT in the cylinder. The filter paper is

"Whatman No. 17" with a filtration area of 12.5 cm^2. 100 grams of fine tailings is placed in the cylinder. After the pressure is applied, the filtrate (water) is drained out from the bottom of the cylinder and collected in a beaker placed on a balance (PM 2000, Fisher Scientific). The balance is interfaced to a micro computer which recorded the weight of the filtrate as a function of time (50-second time interval for the tailings). Each filtration test was done in 5000 seconds and a total of 100 data points were collected. The viscosity of filtrate is measured using a capillary viscometer at the same temperature as the filtration temperature. The weight of the filtrate at time t is then converted to volume. If the density of the filtrate is unknown a separate measurement of the density is required. A graph of time/volume (t/V) *versus* volume (V) is plotted. The slope of the resulting straight line is then used in the calculation of SRF. All pressure filtration experiments should be conducted at the same temperature.

4.7.3 Results

A large body of experimental data for the specific resistance to filtration for Syncrude and Suncor tailings has been reported by Xu and Mikula [1,2]. They studied the effects of acid-base treatment (pH), calcium ion addition, and bicarbonate concentration on the SRF of the fine tailings [1]. They also studied the SRF behavior of fine tailings generated from the CHWE and OHWE processes [2]. Experimental results indicated that SRF is a very useful index test for characterization of fine tailings. Figure 4.7-1 shows the SRF values for Syncrude and Suncor tailings as a function of pH. At low pH or high pH the SRF, and thus filterability, was reduced significantly. The distinct correlation between SRF and pH of the tailings agrees with correlations of other properties of the tailings such as the elastic modulus, electrosonic amplitude (ESA), volume of centrifuged water, *etc.* [3]. In other words, a peak or a valley in the correlation of those properties with pH was also observed in the same pH range as that of SRF.

The addition of calcium ions into MFT enhances the flocculating effect on fine particle dispersions due to the effect on the electric double layer. This leads the fine particles to approach more closely and flocculate. As a result, the filterability of the MFT is improved by the addition of calcium ions. Xu and Mikula [1] found that the SRF values for MFT treated with $CaCl_2$ and $CaSO_4$, as expected, were considerably smaller than values for the original tailings, and that there was no difference between $CaCl_2$ and $CaSO_4$ treatments, indicating the effect of calcium solely.

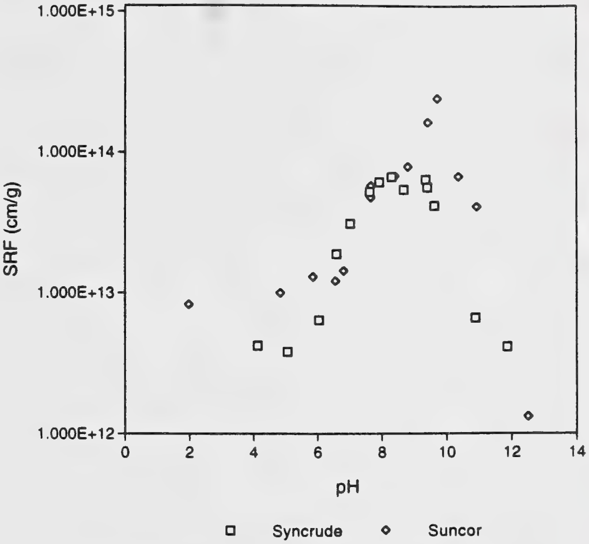

FIGURE 4.7-1. Specific resistance to filtration for Syncrude and Suncor MFT as a function of pH. The maximum resistance to filtration occurs near the natural pH of the tailings.

By studying the SRF of tailings generated by the CHWE and OHWE processes, a noticeable difference was found between the CHWE and OHWE tailings, indicating the effect of the process or water chemistry on MFT properties [2]. Figure 4.7-2 displays SRF values for these tailings as a function of pressure. It shows that the filterability of the OHWE tailings is better than that of CHWE tailings. This is also in agreement with the results from the other index tests [4,5].

4.7.4 References

1. Xu, Y. and Mikula, R. "Filterability and surface tension of mature fine tailings: the effect of pH and calcium ions," *Division Report* WRC 93-37 (CF), CANMET, Energy, Mines and Resources Canada, 1993.
2. Xu, Y. and Mikula, R. "Filterability of oil sands fine tailings," *Division Report* WRC 94-55 (CF), CANMET, Natural Resources Canada, 1994.
3. Mikula, R., Angle, C.W., Zrobok, R., Kan, J., and Xu, Y. "Factors that determine oil sands sludge properties," *Division Report* WRC 93-40 (CF)., CANMET, Energy, Mines and Resources Canada, 1993.

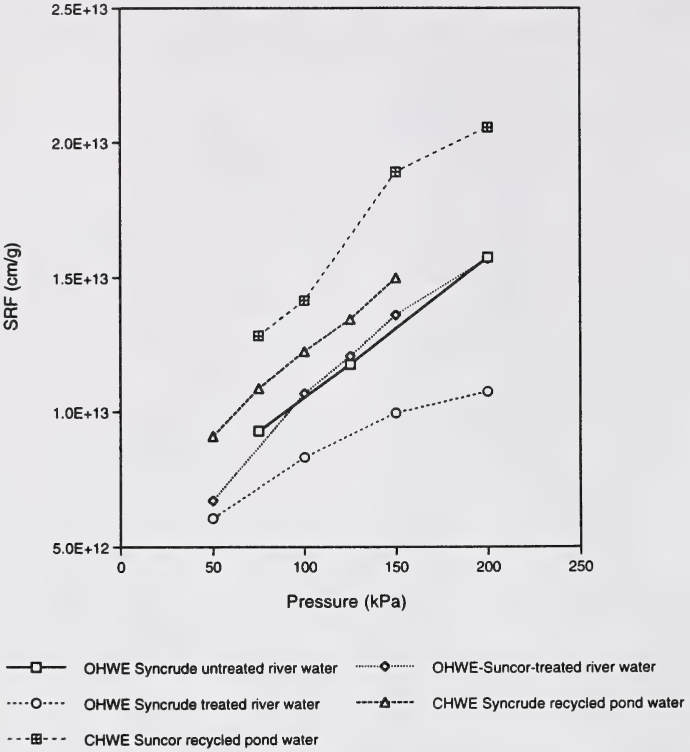

FIGURE 4.7-2. Specific resistance to filtration a function of pressure for five tailings samples. For a given ore, the Clark tailings have a higher SRF, indicative of a more disperse clay suspension.

4.8 Part 4: Low-Speed Centrifugation

4.8.1 Frozen-Image Centrifuge

The Trition – W.R.C. Frozen Image Centrifuge Type TW161 was designed by the Water Resources Centre, Stevenage, U.K., to perform sludge thickenability tests [1]. The method was adapted as an index test for fine tailings characterization.

4.8.2 Methodology

The centrifuge accommodates two samples and incorporates a stroboscope synchronized to the speed of the centrifuge to freeze the image of one of the samples for observation. The fall of the interface between the sludge and the supernatant liquor in the centrifuge tube can thus be monitored. A switchable four-digit LED display indicates either the rotational speed of the sample tubes or the duration of the test. When the test has been completed, the test duration is held in memory until the centrifuge is switched off. Three speeds were used in this experiment: 600, 900, 1185 rpm. The corresponding centrifugal accelerations are 59g, 133g, and 230g. Settling interface levels were recorded as a function of time. This is done by viewing the tube samples through the window of the centrifuge lid while engaging the stroboscope. The length of a test run may be up to 600 minutes, depending on the sample, to allow a sample oil sand tailing to reach a final stable compactness.

The tube is 100 mm long and 17 mm in outside diameter. Before pipeting the tailings samples into the test tube, the samples were inverted 60 times in a minute to achieve the desired dispersion of the sample.

4.8.3 Results

An investigation was carried out into the accelerated settling behavior of five tailings samples. The five tailings samples are composed of the beach runoff samples from the OHWE/CHWE comparison test. The frozen-image centrifuge was used to obtain the sedimentation curves or change of MFT volume with time.

Figure 4.8-1 shows the change of MFT sample volume as a percentage of initial volume with time under 230g centrifugal acceleration (1185 rpm) in the frozen image centrifuge. It compares the Suncor and Syncrude OHWE tailings with their corresponding CHWE tailings. OHWE tailings settle much faster than CHWE tailings. At 100 minutes, the various beach runoff samples could already be ranked in terms of settling as:

Suncor OHWE, > Syncrude OHWE, treated river water > Syncrude OHWE, untreated river water > Syncrude CHWE > Suncor CHWE.

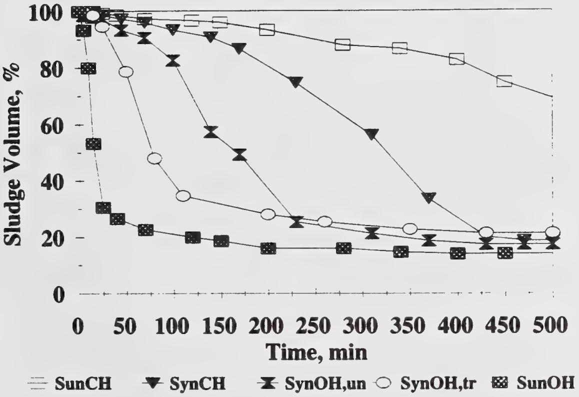

FIGURE 4.8-1. Low speed centrifugation tests of five tailings samples. These test results correspond to those obtained from conventional settling under 1g. The OSLO (OH) process tailings settled much faster than the Clark (CH) tailings from the same ore.

After 500 minutes, all of the samples except for SunCH had sedimented to ~20% of their original volumes. This behavior mimics the long-term settling behavior determined from 1g settling tests. Both the 230 g settling index test and the 1g settling test indicate that all the samples eventually sediment to the similar settled volumes and solids content.

Accelerated settling of the oil sand tailings displays the same three stages of settling as the ordinary settling of tailings under gravity, namely: the induction period; the settling period when quick settling takes place; and the thickening period, when very slow changes can be measured by the thickening of the sediment. OHWE tailings show no induction time period, but in other respects they are similar. Experimental results indicate that the diluted CHWE tailings have shorter settling periods. The 2.5 wt% solids CHWE tailings has a settling period of only half that of the original 8.35 wt% CHWE tailings. When the OHWE tailings solids sediment at higher centrifugal forces, they do not show such a distinct dependence upon initial solids concentration.

There is no noticeable difference in the primary settling rate of Suncor OHWE tailings as a function of centrifugal force, *i.e.*, 230 g, 173 g, and 115 g, respectively. This is because the observable sedimentation is controlled by the particle size distribution in the submicron range and settling is directly proportional to g force but inversely proportional to particle size (radius). With the relatively coarse Suncor OHWE tailings it is difficult to distinguish between the effects of various g forces. For CHWE tailings, a higher measured centrifugal force decreases the settling time.

4.8.4 Reference

1. Kan, J. and Mikula, R. "Particle size distribution and sedimentation of five OHWE and CHWE tailings," *Division Report* WRC 94-51 (CF), CANMET, Natural Resources Canada, July 1994.

4.9 Part 5: Double Hydrometer Test

4.9.1 ASTM Hydrometer Method

An ASTM 152H hydrometer was used for the particle size analysis and to characterize the degree of aggregation of the clays.

4.9.2 Methodology

The specifications and dimensions of the hydrometer used can be found in the Annual Book of ASTM Standards [1]. The ASTM standard test method [2] for particle size analysis was applied in the experiment. It was noticed that particles in the MFT samples were flocculated. In order to measure their actual size and size distributions without any change in the tailings, no dispersant was added to the tailings, except in the later part of the experiment, when dispersant was added to observe its effects. Sodium hexametaphosphate and sodium bicarbonate were used as dispersants. The hydrometer method used in the particle size analysis of the tailings is based on the ASTM Designation D 422-63, standard test method for particle size analysis of soils. The particle size test involves sampling of the five different types of MFT: SynCH; SunCH; SynOH, tr; SynOH, un; and SunOH. All five samples were diluted using their original waters to approximately 5% and 2.5% solids by weight and placed into 1-L graduated cylinders. The samples were dispersed by repeatedly inverting the graduated cylinder for 1 minute or until all of the solids were in suspension. The suspension was then allowed to settle and hydrometer readings were taken at specified time intervals. The test was terminated when the hydrometer reading became relatively unchanged with time. Generally, the time required for one test ranged from one day to two or three weeks.

Modifications to the ASTM standard method used during the particle size measurement of the tailings are summarized as follows: The tailings were not air dried before they were tested. As previously mentioned, a dispersing agent was not used. The tailings were left in their natural settling medium and were not placed in distilled water. The tailings were dispersed by repeatedly inverting the graduated cylinder. The composite correction factor (discussed further below) was determined by allowing the hydrometer to come to an equilibrium level in 1 L of the same tailings water which, as described above, was obtained from the water in the settling tank after being filtered by a 1-micron filter paper.

The principle of particle size analysis using an ASTM hydrometer is discussed briefly in this section and details can be seen in references [2,3]. The hydrometer is designed to measure the density of a solid suspension. Assume that particles in the suspension are fully dispersed and settle according to Stoke's equation. That is, the time required for a particular particle to settle down a specified distance can be calculated. In other words, after a certain period of settling, only those particles smaller than a certain value will stay in the suspension while larger ones will have already passed the measurement section. Thus by measuring the changing density of the suspension with time, the size and the corresponding percentage of finer particles remaining in the suspension can be determined.

The diameter of a particle corresponding to the percentage indicated by a given hydrometer reading can be calculated according to Stokes' law, assuming that a particle of this diameter was at the surface of the suspension at the beginning of sedimentation and had settled to the level at which the hydrometer is measuring the density of the suspension:

$$D = K\sqrt{\frac{L}{T}}$$

where:

D = diameter of particle, mm.

K = constant depending on the temperature of the suspension and the specific gravity of the soil particles. Value of K can be found in Table 3 of the ASTM standard method D 422.

L = distance from the surface of the suspension to the level at which the density of the suspension is being measured, cm. This distance is known as effective depth. Values of L can be found in Table 2 of the ASTM standard method D 422.

T = interval of time from beginning of sedimentation to the taking of the reading, min.

The percentage of soil remaining in the suspension at the level at which the hydrometer is measuring the density of the suspension may be calculated as follows: For hydrometer 152H:

$$P = (Rc \times a) / w * 100$$

where:

P = percentage of soil remaining in suspension at the level at which the hydrometer measures the grams per litre of solids in suspension, %.

Rc =corrected hydrometer reading using the composite correction factor, g/L. Values of the correction factor can be found in section 7 of the ASTM standard method D 422. The ASTM hydrometer is calibrated at 68°F (20°C) distilled water or demineralized water. The density of the tailings water is greater and a correction factor is needed.

a = correction factor based on the specific gravity of the tailings tested. Values of A can be found in Table 1 of the ASTM standard method D 422.

w = dry weight of the tailings that were tested in 1 L of suspension, g.

4.9.3 Results

An investigation was carried out into the particle size and size distribution of five tailings samples. The five tailings samples are composed of the beach runoff samples from the OHWE/CHWE comparison test. An ASTM hydrometer test was used to measure the particle sizes and size distributions of the tailings samples and their corresponding settling behavior. The effects of solids content, dispersants, and their concentrations on particle sizes and size distributions were investigated. Technical issues in applying the ASTM hydrometer method to the measurement of fine tailings are discussed and detailed results can be found in CANMET Division Report WRC 94-51 (CF).

The particle size distributions of the five beach runoff samples diluted to 2.5% solids (with no dispersant added) using their own waters is shown in Figure 4.9-1. The five distribution curves show pronounced differences for small particle sizes. For particle sizes larger than 5 microns, all of the five distribution curves converge. In the case of suspensions with 2.5 wt% solids, particle sizes larger than 35 microns account for only about 2% of the total solids in the five samples. The Suncor CHWE beach runoff sample had the largest proportion of the smallest size particles: 45% of the particles are smaller than 1 micron; this is followed, in order, by Syncrude CHWE and Syncrude OHWE (treated water).

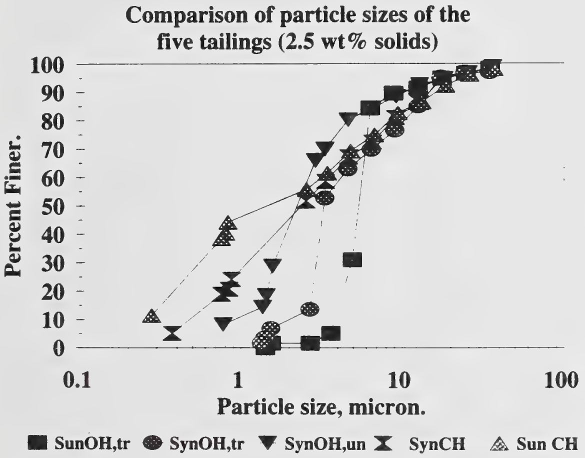

FIGURE 4.9-1. Particle size distributions for five tailings samples. The Clark (CH) tailings have a significantly smaller size distribution than the OSLO (OH) tailings from the same ore. The tr means the process water was treated to increase the calcium level. By repeating these tests with a dispersant and comparing the particle size distributions, a degree of aggregation index can be defined.

A second series of hydrometer tests was conducted using deionized water and a chemical dispersant. The dispersant chosen in this experiment was 2500 ppm sodium hexametaphosphate. Additions of large amounts of dispersant had no effect on reducing the particle sizes in Suncor CHWE tailings, indicating that the Clark tailings are already dispersed (Figure 4.9-2). However, a dispersant added to Suncor OHWE tailings resulted in a significantly smaller measured size distribution, similar to that observed for the Clark tailings (Figure 4.9-2). The difference in the percentage of particles less than 2 microns between the "neat" and dispersed hydrometer tests can be defined as the degree of dispersion or the degree of aggregation of the tailings. The double hydrometer test (dispersed and neat) is a useful index test to quantify the degree of aggregation of the clays in a tailings sample.

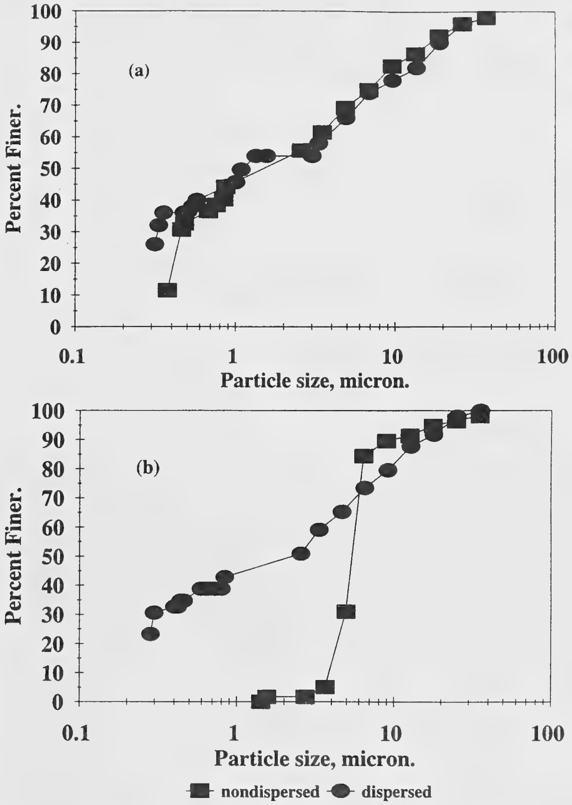

FIGURE 4.9-2. Particle size distributions for Clark and OSLO tailings in the tailings water and with a dispersant. The Clark tailings are already dispersed, so the addition of dispersant results in no decrease in particle size. With the dispersant, the OSLO tailings aggregates are broken up, resulting in a significantly smaller size distribution. The degree of aggregation can be defined by the difference in the percentage of particles smaller than 2 microns with and without dispersant.

4.9.4 References

1. "Standard specification for ASTM hydrometers, ASTM designation: E 100–81," Annual Book of ASTM Standards, 74-82, Vol. 05.03, 1991.

2. "Standard test method for particle-size analysis of soils, ASTM designation: D 422–63," Annual Book of ASTM Standards, 90-96, Vol. 04.08, 1991.
3. Allen, T. "Particle size measurement," New York, Chapman and Hall, 310–343, 1990.
4. Kan, J. and Mikula, R. "Particle size distribution and sedimentation of five OHWE and CHWE tailings," Division Report WRC 94-51 (CF), CANMET, Natural Resources Canada, July 1994.

4.10 Part 6: Microscopy

4.10.1 Objective

The miscroscopic methods were developed to relate the floc structure to bulk MFT properties. Three techniques were used, including light microscopy, confocal microscopy and scanning electron microscopy.

4.10.2 Methodology

4.10.2.1 *Reflectance Measurements Using Light Microscopy (LM)*

The indices of refraction and absorption are important physical constants used to describe optically transparent materials. In the case of opaque materials, the reflectivity or reflectance is measured, and is related to the refraction and absorption indices by the Beer-Fresnel equation:

$$R = \frac{Ir}{Ii} = \frac{\left(n - n^0\right)^2 + n^2 k^2}{\left(n + n^0\right)^2 + n^2 k^2}$$

where R is the reflectance, Ir is the intensity of the reflected beam, Ii is the intensity of the incident beam, n and k are the indices of refraction and absorption of the sample, and n^0 is the index of refraction of the immersion medium.

One implication of the Beer-Fresnel equation is that the reflectance increases with the indices of refraction and absorption. The index of refraction is influenced by the density, molecular weight, and molecular structure of the material under study. Calculation of the indices of refraction and absorption for opaque substances can be performed by measuring the reflectance of the material in air (or water) and then using immersion oil. The two indices, n and k, can be calculated by solving a system with two independent equations, one for air and one for oil [1–7].

The dependence of the reflectance on the indices of refraction and absorption, and density can be used to assess the effect of various treatments on MFT. The examination of these suspensions by light (LM) and confocal laser scanning microscopy (CLSM) has demonstrated that the inorganic components form ordered clusters of a wide variety of sizes. Their reflectance values can change due to variations in the indices of refraction and absorption, which are caused by differences in the interparticle ordering and water chemistry. A significant number of ordered clay clusters can show high reflectance values and the difference between the reflectance of the aggregates and the areas without aggregates may be large. It can be inferred that the effect of chemicals causing dispersion of the aggregates reduces both the mean and the range of the reflectance values for the suspensions. The reflectance values measured by LM can be characterized in terms of the mean values and the variance. However, a representative number of data points must be obtained in order to correlate the effect of chemical and/or physical treatments with changes in the optical properties.

A Carl Zeiss research microscope photometer with an incident light system (reflected light) was utilized for the measurement of the reflectance values of the suspensions. A combination of a photomultiplier and a monochromator was used to measure the intensity of the reflected light at 545 nm. Data acquisition and photomultiplier output calibration were done with a microcomputer interfaced with the photomultiplier. The system was calibrated with a silicon carbide standard of a reflectance of 7.46% in oil. The reflectance of the standard used should be similar or higher than that of the sample to be measured. A measuring diaphragm 3 microns in diameter and having a magnification of 600X is suitable for measurements performed under bright field illumination or white light, in which the incident light is not polarized.

To be considered statistically representative, a minimum of 99 reflectance values should be measured using a 0.2-mm distance between the points across the surface. Sample movement can be controlled by an automatic point counter (Swift Model F 415C). As each field comes into view, the reflectance of sample area immediately under the measuring diaphragm is recorded. Another application of reflectance measurements is the rank determination of coal [8–11]

The MFT samples should be gently mixed to ensure homogeneity. This is done by slowly inverting the container 20 to 30 times and allowing a standing time of 10 minutes before collecting the sample for LM. The samples are taken from the central part, near the bottom of the containers, using a wide-mouth Pasteur pipette. The free water and the fines on the top of the containers can be avoided by applying slight pressure with a rubber bulb. This is done to collect the samples in the most consistent way for comparison of their microscopic properties. Afterwards, the sample is deposited in the chamber (0.3 mm depth, 7.3 mm width, and 30 mm length) of a stainless steel sample holder, covered with a cover slip, and examined using oil immersion objectives.

4.10.2.2 *Confocal Laser Scanning Microscopy (CLSM)*

In the CLSM technique, sample components are scanned point by point with a focused laser beam. The reflected or emitted light (fluorescence) from the specimen is detected by two photomultipliers, digitized, and displayed on a monitor. The main feature of CLSM is that it removes out-of-focus information from the image by means of adjustable pinhole irises placed before the detectors. This permits independent imaging of structures, permitting profiles, three-dimensional reconstructions, and quantitative measurements of height.

CLSM allows for simultaneous acquisition of images in two wavelengths, exciting the fluorescence of the bitumen with blue light (488 nm) and detecting the fluorescence image in the green region (514 nm), while simultaneously detecting, in the second photomultiplier, the inorganic components which show strong reflection (of wavelengths such as 647 nm). The image monitoring screen is split during this mode of acquisition. Image processing techniques allow one to merge the two images to facilitate determination of the association between fluorescent and non-fluorescent sample components. Further details of this technique are given elsewhere [12–15].

Confocal microscopy at WRC is performed using a Bio-Rad MRC-600 imaging system coupled to a Nikon Microphot 2 light microscope. The system is equipped with a krypton/argon mixed-gas laser (15 mW) which can provide lines at 488, 568, and 647 nm. The use of suitable filters allows one to select one of these wavelengths or any combination.

The images for the comparison of the morphology of the samples (Tables 4.10-3 and 4.10-4) were obtained using the simultaneous acquisition mode, in which the image monitor is split to display the reflected and fluorescent images of the same field of view. The samples were examined under the same instrument conditions which provided a magnification of 650X with a theoretical resolution of 0.2 microns on the confocal micrographs.

The MFT samples for morphology comparison were taken from the central part, near the bottom of glass containers, using a wide-mouth Pasteur pipette. The free water and the fines at the top of the containers were avoided by applying slight pressure with a rubber bulb. This was done to collect the samples in the most consistent way in order to compare their morphologies. Afterwards, the sample was deposited in the chamber (0.3 mm depth, 7.3 mm width, and 30 mm length) of a stainless steel sample holder, covered with a cover slip, and examined using oil immersion objectives.

Microscopic examination of the samples was performed using blind experiments in which the microscopist was not informed of the settling behavior of the samples. The microscopic comparisons, such as the effect of OHWE *versus* CHWE on the microscopic characteristics of the beach runoff samples, were made by preparing two samples in identical sample holders, prior to side-by-side microscopic examination. About 100 fields of view were examined by LM to assess differences in the morphology of the samples. Particular attention was given to the aggregation of clays and the size of the dispersed oil (droplets *versus* globules). It should be pointed out that the qualitative assessment of the samples proved valid and that the differences between the samples was performed by direct comparison. However, this method depends on the experience of the microscopist, and a less subjective evaluation using image analysis would be necessary to provide numerical data. Such a numerical comparison would provide size and shape factors for the clay aggregates and dispersed bitumen, as well as their relative concentration.

4.10.2.3 *Scanning Electron Microscopy*

The scanning electron microscope used was a Hitachi X-650 equipped with both energy-dispersive (EDS, 30 mm^3 Si(Li) detector; TN5402) and wavelength-dispersive spectrometers. The EDS on the Hitachi X-650 has a Z-max window which allows for the detection of carbon and oxygen in the samples. The SEM was also equipped with a Hexland DN302 cold stage used for examining frozen samples. The rapid freezing of the samples maintains the morphology or the relationship between various components. By keeping the sample frozen on a cold stage in the electron microscope, it is possible to image the fine tailings and also to get compositional information from the x-rays emitted as the electron beam strikes the sample. In samples with high water contents such as these, it is possible to sublime away the water under controlled conditions, in order to better reveal the associations between the various components. The temperature of the cold stage in the electron microscope was maintained at ~90 K by an Oxford ITC4 nitrogen heat exchanger and temperature-controller unit. Each sample was placed onto a copper sample stub and immersed in liquid nitrogen. While frozen, the sample was fractured and subsequently transferred into the SEM chamber for observation. Secondary electron images of the samples were acquired at 15 or 20 kV and stored in the Tracor Northern TN8502 image analysis system.

In these cryogenic techniques, the rate of freezing is critical in minimizing the creation of artifacts [16–25].

Artifacts, or structures in the sample which are not present naturally but only occur due to ice crystal formation during the freezing process, are a particular problem in the freeze fracture technique. Artifacts occur because, at slow freezing rates, ice crystal formation tends to push particles and concentrated solutes into eutectic phases which later freeze, producing features that are not actually part of the sample structure. Ice crystal formation occurs regardless of the freezing rate; however, at higher freezing rates, ice crystals are smaller. One only requires a freezing rate high enough to ensure that ice crystals are smaller than the resolution at which the sample is observed. The freezing rate achieved with a nitrogen slush should produce ice crystals on the order of 50 nm and smaller, although the freezing process is complex and freezing rates are difficult to predict. Liquid nitrogen, nitrogen slush, liquid freon were all tested. No differences were observed in sample morphology, so liquid nitrogen was used as a matter of convenience. Ice crystal formation was found to be well below the resolution of the electron microscope for frozen hydrated samples. Another consideration with sample handling and observation in this mode is the possibility of beam damage and creation of artifacts associated with the large amount of energy per area deposited on a sample when the electron beam is finely focused. This can be minimized by using low beam currents and, in this case, beam damage, even with long observation times, was minimal. The final artifact consideration is the sublimation process. Careful removal of water from a sample by sublimation can produce structural artifacts *via* recrystallization of the water phase if sublimation is carried out at too high a temperature (usually above 220 K). The sublimation temperature need not go this high since significant sublimation rates can be achieved at lower temperatures in the vacuum of the electron microscope. The small sample size used for the cryogenic SEM might result in wall or edge effects that affect the clay structure but the identification of differences in floc morphology with various MFT samples is a valid observation.

4.10.3 Results

The reflectance values determined by light microscopy represent the floc structure. Greater average reflectance means a greater degree of aggregation. The light microscopy results shown in Tables 4.10-1 and 4.10-2 clearly illustrate the changes in floc morphology with water chemistry.

CLSM was used to examine the microscopic morphology of the organic and inorganic components present in untreated as well as treated fine tailings samples at CANMET's Fuel Processing Laboratory. The optical separation of the components achieved with this instrument allowed to study each phase independently and subsequently merge the images to analyze their association.

The effects of several parameters on the microscopic morphology were assessed, including the following:

TABLE 4.10-1. Reflectance measurements for untreated (blank) and treated Syncrude fine tails (99 data points).

Data	Sample identification											
	Blank		HCl to pH 6.0	H_2SO_4 to pH 6.0		HCL to pH 6.0 then $Ca(OH)_2$ to pH 8.0	H_2SO_4 to pH 6.0 then $Ca(OH)_2$ to pH 8.0	$CaSO_4 \cdot 2H_2O$ 1200 pm Ca	$CaSO_4 \cdot 2H_2O$ 800 pm Ca	$CaSO_4 \cdot 2H_2O$ 400 pm Ca	$CaCl_2$ 1000 ppm (ward brine)	$Ca(OH)_2$ to pH 9.0
	Trial 1	Trial 2		Trial 1	Trial 2							
Minimum value	1.00	2.90	1.22	1.36	1.30	1.42	1.40	1.21	1.10	1.14	1.30	1.35
Maximum value	7.19	6.44	4.94	7.00	8.62	374	5.32	7.74	5.28	5.03	2.84	6.55
Mean value (x)	2.94	2.90	2.18	2.63	2.74	2.26	2.18	1.99	1.87	1.85	1.92	2.80
Standard deviation (s)	1.00	0.87	0.57	0.96	1.10	0.42	0.58	0.71	0.65	0.55	0.34	0.83
Coefficient of variation (s/x 100)	34.27	30.17	25.96	36.35	40.18	18.66	26.76	35.88	34.76	29.88	17.82	29.70
Standard error of the mean ($s/\sqrt{n}$)	0.10	0.09	0.06	0.10	0.11	0.04	0.06	0.07	0.06	0.06	0.03	0.08
% Water release (estimate after 1 month)	0		7	8		10	10	11	10	12	17	0

TABLE 4.10-2. Reflectance measurements for untreated (blank) and treated OSLO fine tails.

Data	Sample identification			
	Blank		OSLO fine tails +1500 ppm bicarbonate	
	Trial 1	Trial 2	Trial 1	Trial 2
Data points (n)	99	99	99	50
Minimum value	0.89	0.88	0.12	1.05
Maximum value	6.41	4.72	4.08	2.88
Mean value (x)	2.00	1.94	1.93	1.87
Standard deviation (s)	0.64	0.54	0.50	0.37
Coefficient of variation (s/x·100)	32.17	27.96	25.80	19.81
Standard error of the mean (s/√n)	0.06	0.05	0.05	0.05

1. Effect of the process; comparison of the Clark hot water extraction process (CHWE) and the OSLO hot water extraction process (OHWE).
2. Effect of the ore; Syncrude *versus* Suncor ore.
3. Effect of the water; use of treated river water compared to untreated river water.
4. Effect of $CaSO_4$ addition.

Figures 4.10-1 and 4.10-2 illustrate the differences between the CHWE and OHWE processes regarding the size of the clays aggregates and their association with the dispersed bitumen. Figure 4.10-1 depicts the MFT from the CHWE process while Figure 4.10-2 depicts those from the OHWE process. The micrographs were acquired in the simultaneous acquisition mode with the bitumen phase shown on the left and the inorganic phase on the right. Clearly, the OHWE process produced larger clay aggregates. Results of this type of study are presented in Tables 4.10-3 and 4.10-4.

Figure 4.10-3 shows examples of the changes in floc structure that can be identified between Clark MFT, OSLO MFT and the chemically treated MFT samples that show how floc morphology can be manipulated by adjusting water chemistry [26,27].

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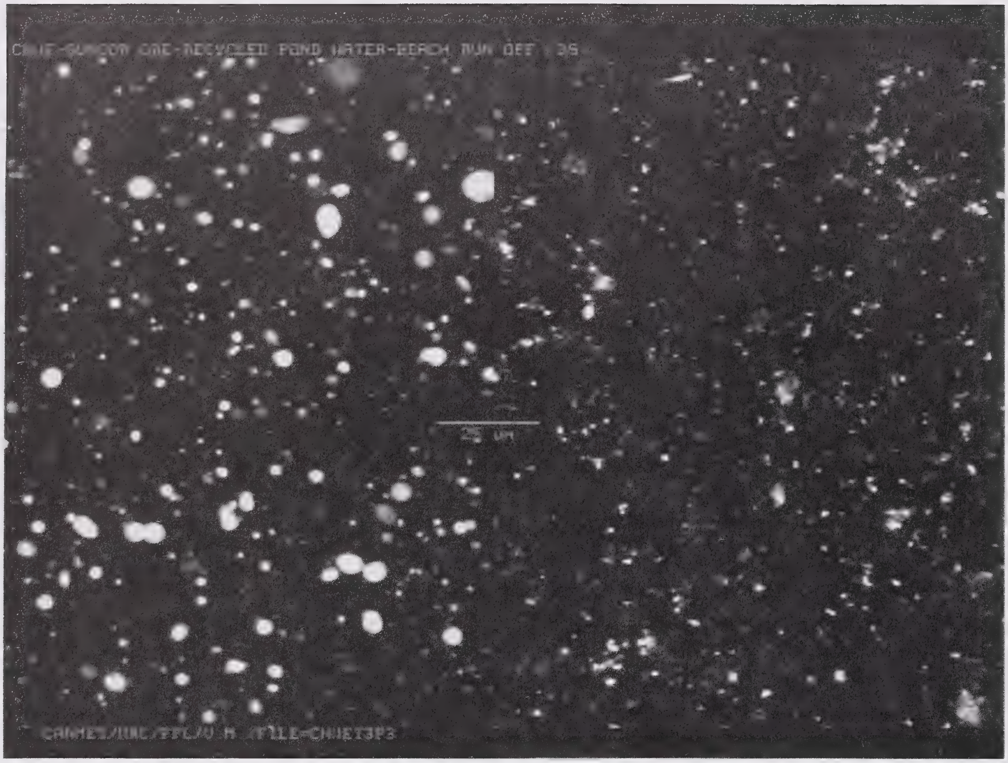

FIGURE 4.10-1. This micrograph of Clark tailings from Suncor ore was acquired using the fluorescent (bitumen image, left) and reflecting (mineral image, right) modes simultaneously. The size of the clay aggregates shown on the right side are significantly smaller than the equivalent OSLO tailings from Suncor ore.

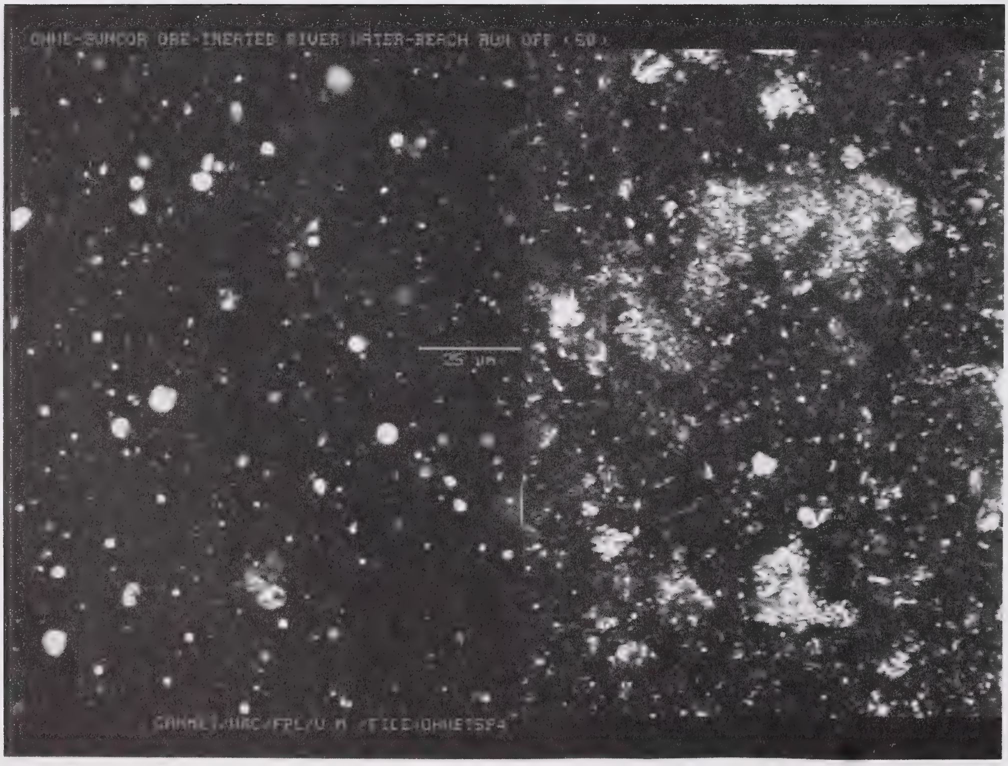

FIGURE 4.10-2. This micrograph of OSLO tailings from Suncor ore was acquired using the fluorescent (left) and reflecting (right) modes simultaneously. The size of the clay aggregates shown on the right side are significantly larger than the equivalent Clark tailings from the same ore.

TABLE 4.10-3. Confocal microscopy of samples vacuum filtered for consolidation testing at the University of Alberta.

	CHWE; Suncor ore; recycled pond water; 32.9% solids	CHWE; Suncor ore; recycled pond water; CaSO4; 31.0% solids	OHWE; Syncrude ore; untreated river water; 31.8% solids
OHWE; Suncor ore; treated river water; 31.3% solids	OHWE: more large clay aggregates and more bitumen globules than CHWE		Syncrude ore: more large clay aggregates than Suncor ore
CHWE; Syncrude ore; recycled pond water; 31.8% solids	Syncrude ore: more large clay aggregates than Suncor ore		OHWE: slightly more large clay aggregates; appears to be associated more with organics than CHWE
OHWE; Syncrude ore; treated river water; 31.0% solids			No significant difference
CHWE; Syncrude ore; recycled pond water; CaSO4; 31.5% solids		Syncrude ore: more large clay aggregates Suncor: some large oil layers associated with inorganics	

TABLE 4.10-4. Confocal microscopy of samples treated at WRC.

	OHWE; Syncrude ore; treated river water; beach runoff	CHWE; Suncor ore; recycled pond water; beach runoff
CHWE; Syncrude ore; recycled pond water; beach runoff	OHWE: more large clay aggregates than CHWE	Syncrude ore: more large clay aggregates than Suncor ore
OHWE; Syncrude ore; untreated river water; beach runoff	Treated river water: more large clay aggregates than with untreated river water	
OHWE; Suncor ore; treated river water; beach runoff	Syncrude ore: more large clay aggregates than Suncor ore	OHWE: more large clay aggregates than CHWE

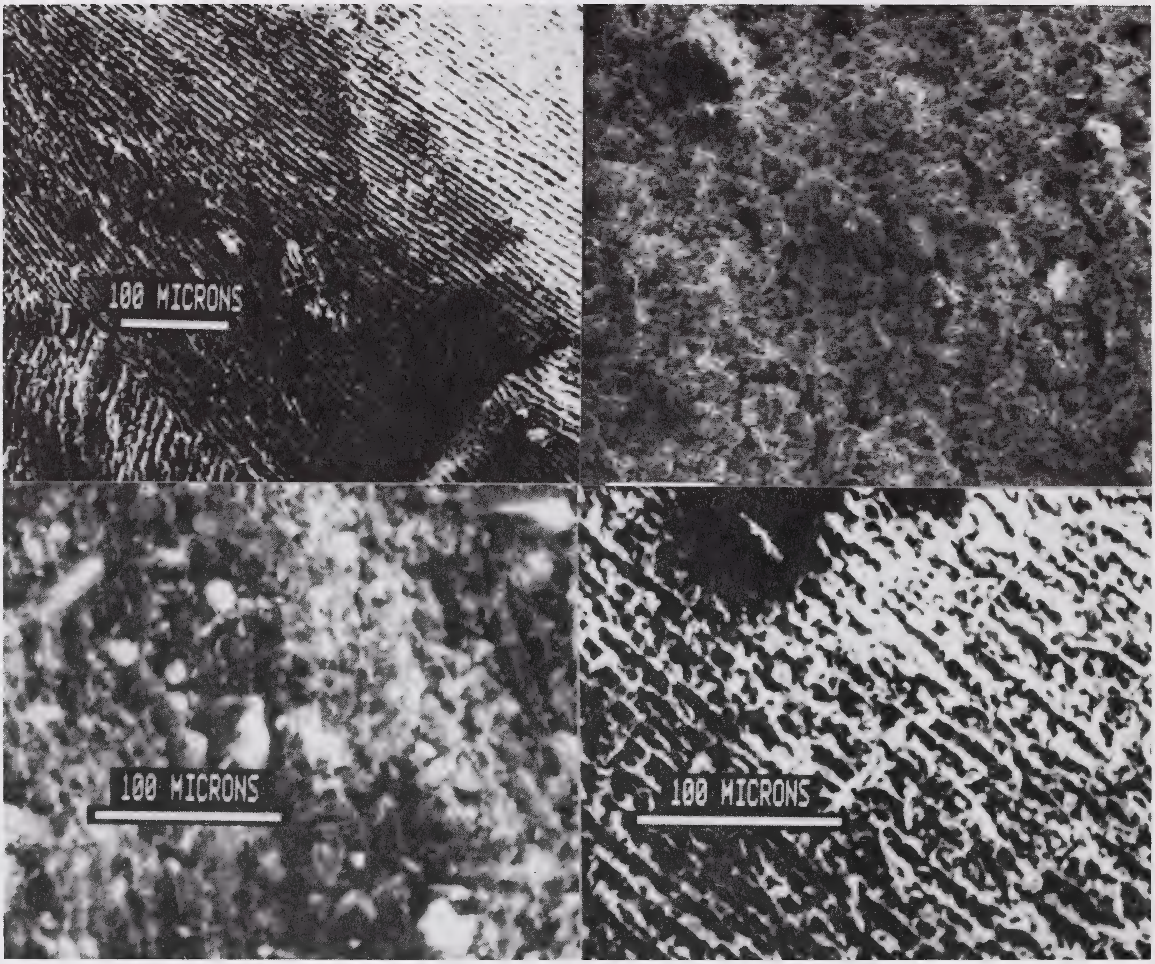

FIGURE 4.10-3. Scanning electron micrographs of the differences in floc morphology for Clark and OSLO MFT as water chemistry changes. The top left photograph is of Clark MFT showing the distinctive edge to edge dominated floc structure. The top right photograph is OSLO MFT which shows a more random clay association and a clearly different floc structure. The bottom left photograph is a chemically treated Clark MFT which now resembles the OSLO material. The bottom right photograph is OSLO MFT treated with 1500 ppm bicarbonate and it now has a structure similar to the untreated Clark MFT. These microscopic differences correspond to differences in the bulk behavior (as determined from the index tests).

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5.0 GEOTECHNICAL PROPERTIES OF MATURE FINE TAILINGS (MFT)

5.1 Introduction

Fine tailings is the fine grained slurry which results after the coarser sand grains have segregated out of the plant tailings after discharge from the tailings pipe during either cell construction or beach operations. The fine tailings has the consistency and behavior of a heavy fluid.

The surface of the fine tailings deposit has a solids content typically in the order of 10% to 15% (weight of dry solids divided by the total weight). In general, the fine tailings will consolidate to a solids content of close to 30% over a period of several years after deposition. After this, it has been observed that the rate of solids content increase (consolidation) is substantially reduced. The material with a solids content greater than 30% is termed mature fine tailings (MFT).

The fine tailings and even mature fine tailings straddles the boundary of fluid and soil. After hindered settlement has taken place, the particles are in contact with one another and effective stresses can be transmitted from one particle to another. At this point, the soil skeleton is, technically speaking, a soil and therefore soil mechanics concepts should apply. However, before this point the material appears and behaves as a fluid. The actual transition from fluid behavior to soil behavior is not distinct.

5.2 Characterization of MFT

MFT is comprised of mineral clays and silts, bitumen and water. Figure 5-1 shows a typical grain size distribution of the mineral grain portion of MFT. MFT at the Syncrude site typically has only 3% to 5% sand sized particles. Samples obtained from Suncor in Pond 1 have as much as 25% sand. The additional sand maybe from particle migration since the pond is much older than Syncrude's pond.

The clays are predominantly kaolinite (80%). Other clay minerals include illite (15%), montmorillonite and chlorite (Roberts, Yong and Erskine, 1980). Kaolinite is typically one of the least active (in terms of affinity for water) clay minerals.

Bitumen contents (defined as weight of bitumen divided by weight of mineral grains) vary from 2% to 30% but is probably more typically in the range of 2% to 10%. As discussed in the previous section, the solids content of MFT varies from 30% to 60%.

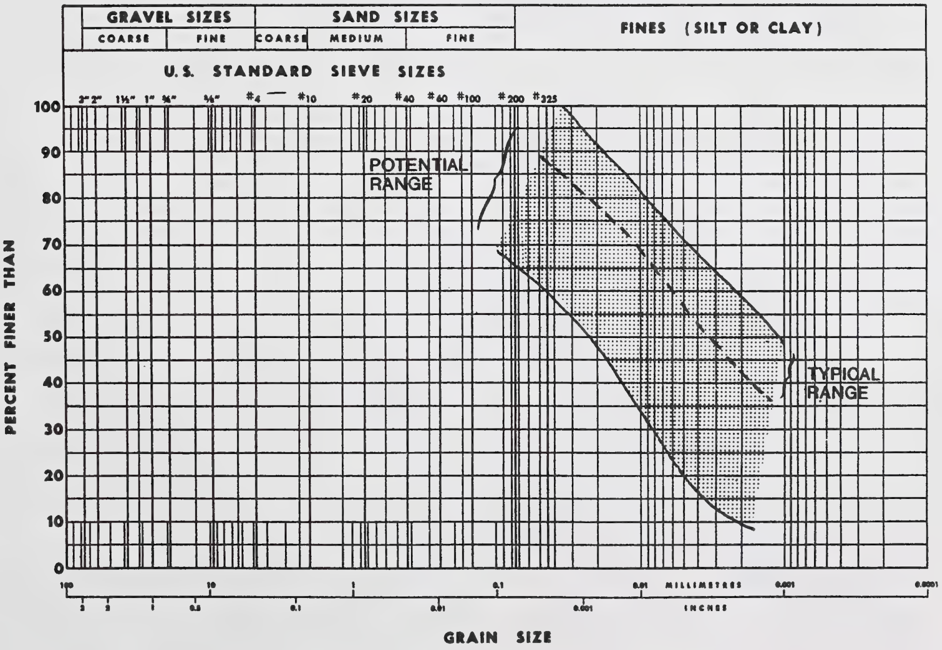

FIGURE 5-1. Typical grain size distribution of oil sands fine tailings.

The liquid limit of MFT is the range of 40% to 75%. The plastic limit is in the range of 10% to 20%. These Atterberg Limits are shown with Atterberg Limits from other fine grained mine waste slurries on Figure 5-2.

The fine grained slurry behaves as a viscous fluid with a finite yield strength. The behavior of fine tailings can be modelled as a Bingham plastic or a pseudo-plastic material. Clearly the yield strength and viscosity vary with solids content. The yield strength of MFT varies from 0 to 520 Pa. Viscosity can vary from 0 to 5000 cP. The viscosity of MFT varies with time. This time dependent behavior is termed thixotropy.

5.3 Compressibility

The compressibility of a soil describes the amount that a soil skeleton will compress under a given load. It is the derivative of a relationship between load and volume. Typically this relationship is given in terms of void ratio (volume of voids divided by volume of solids) *versus* effective stress. Note that this relationship is independent of time.

A typical compressibility curve for MFT is given in Figure 5-3. A compressibility curve for a 20% fines sand/fine tails mixture is also shown on the plot for comparison. Note that the relationship between void ratio and effective stress is highly non-linear as the material becomes less compressible as the void ratio decreases.

The difference between the two curves indicates that even when fully consolidated, the fine tails consistency at low effective stresses (*i.e.*, near the surface) is still very thin and unable to support any load. For example, at a depth of 3 m, the solids content of fine tails, when it finishes consolidating, will only be approximately 60%. However, the sand/fine tails mixture shown on Figure 5-3 could have a solids content of close to 80% at a depth of 3 m after consolidation.

5.4 Hydraulic Conductivity

The hydraulic conductivity (or permeability) of a soil is the property which determines the rate of flow a fluid through a soil under a given hydraulic gradient. Consequently, hydraulic conductivity determines the rate consolidation as opposed to the amount. For a given soil, the hydraulic conductivity is a function of the void ratio. Since the void ratio varies greatly in a consolidating fine tailings deposit, it is necessary to know the hydraulic conductivity over a range of void ratios.

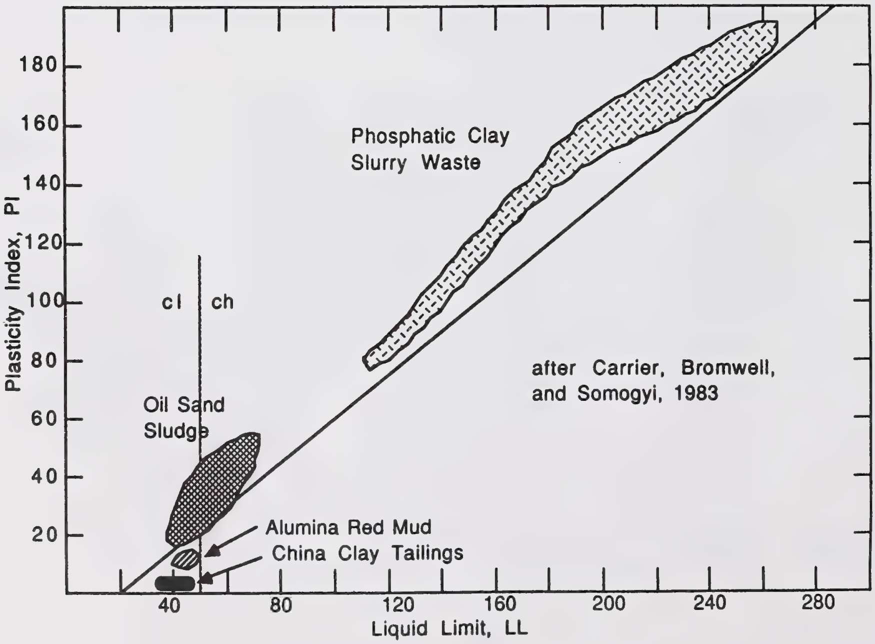

FIGURE 5-2. Plasticity of various mine waste sludges.

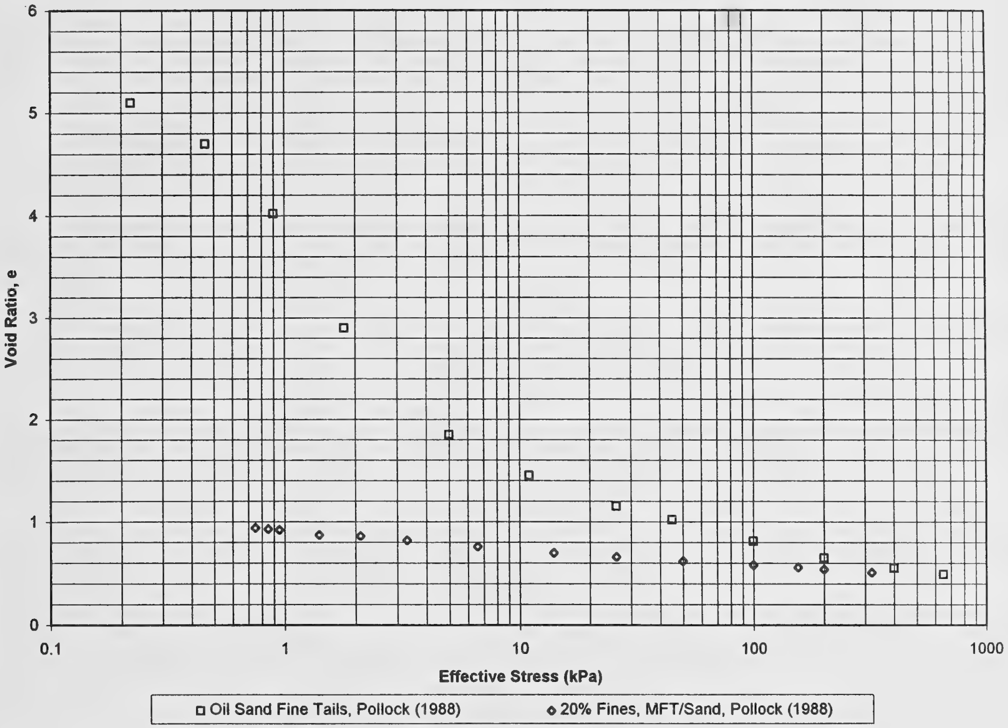

FIGURE 5-3. Compressibility of oil sand fine tails.

Figure 5-4 shows a typical hydraulic conductivity *versus* void ratio relationship for MFT. Note that the hydraulic conductivity varies over 5 orders of magnitude and that the relationship is highly non linear. A certain amount of scatter exists at the high void ratios (*i.e.*, low solids contents) due to the difficulty of obtaining hydraulic conductivity for low solids content fine tails.

Although the initial hydraulic conductivity of MFT is only slightly lower than that of beached tailings sand (10^{-3} to 10^{-4} cm/s), the hydraulic conductivity of MFT decreases substantially with decreasing void ratio (or increasing solids content). This sensitivity of hydraulic conductivity to solids content implies that the rate of consolidation decreases noticeably as consolidation proceeds.

This sensitivity of hydraulic conductivity to solids content has another implication at the base of a pervious pond or column. The fine tails becomes so impermeable at the pervious base, due to the increase of solids content, that the fine tails virtually seals off the pervious base and the pond or column very quickly behaves as if there is only one way drainage (*i.e.*, to the surface).

5.5 Strength

Strength tests which have been conducted on MFT consist primarily of vane shear tests, although some falling cone tests and expanding fluid sphere tests have also been conducted.

Typical values which have been reported vary from 0.2 kPa to 2.5 kPa. As would be expected, these values are very low relative to typical geotechnical ranges for consolidated soils. This finding is consistent with the compressibility of MFT as discussed in Section 3.0.

The implication of the low strength is that the fine tails, even after consolidation, is unable to support any construction traffic that would be required to reclaim the surface. Therefore, to reclaim the land, it would be necessary to increase the solids content of the MFT near the surface beyond what self weight consolidation will be able to do so that it could support traffic. Possible solutions include:

- Providing an initial blanket surcharge (possibly with sand) to the surface of the fine tails. This would require sophisticated spreading techniques and the surface may not even support a small surcharge.

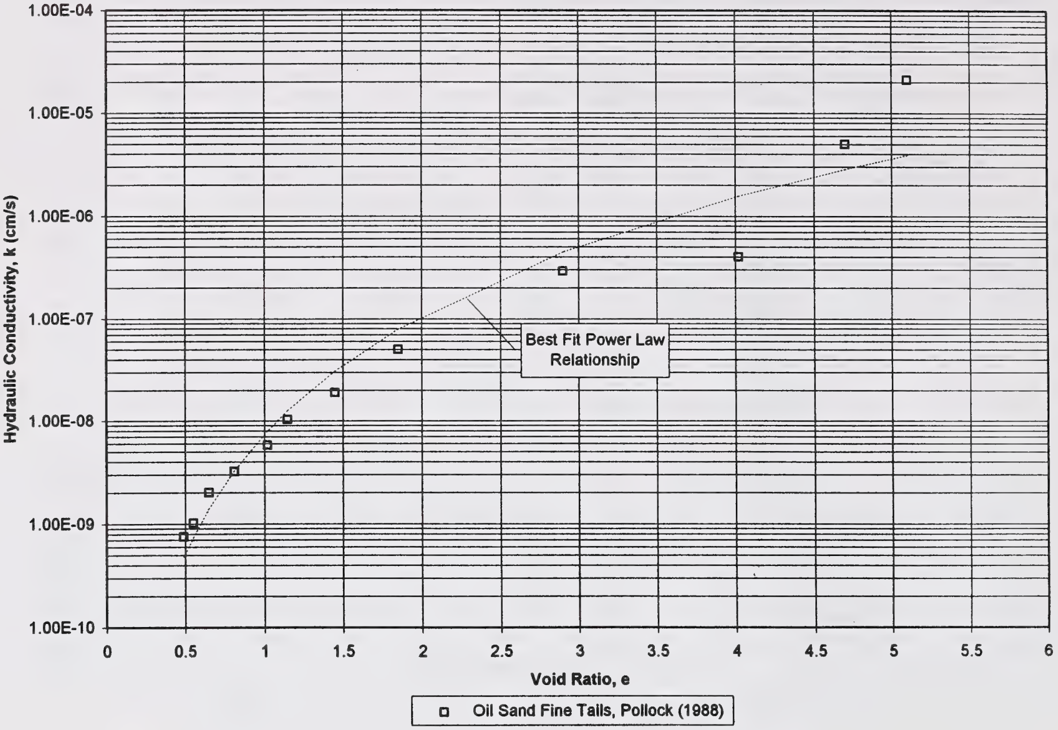

FIGURE 5-4. Permeability of oil sands fine tails.

- Drain the surface and allow natural processes such as drying and freeze thaw to dessicate the material (Sego and Dawson, 1994).
- Combination of the above two.

Work done by Banas (1991) has shown that the strength of fine tails depends on time (independent of consolidation). This dependency on time is termed thixotropy. However, the increase in strength is not enough for purposes stated above.

5.6 MFT Consolidation Predictions

5.6.1 Effective Stress Approach — Large Strain Consolidation

Consolidation of soil is traditionally determined using Terzaghi's classic theory which results in an equation similar in form to the heat flow equation. However, in order to achieve this mathematically convenient equation, some assumptions were required such as: hydraulic conductivity is constant, the relationship between effective stress and void ratio is linear, and self weight of the soil is ignored.

While those assumptions (and others) are adequate for soils typically dealt with in geotechnical practice, they are, as shown in the previous sections, completely invalid for slurry consolidation. Therefore Gibson, England, and Hussey (1967) extended the consolidation theory to allow for non-linear hydraulic conductivity and compressibility relationships as well as accounting for self weight of the soil. This theory is termed Finite Strain or Large Strain Consolidation Theory.

Pollock (1988) developed a computer program based on Finite Strain Theory in order to model the consolidation of slurries such as oil sand fine tails. A series of consolidation and permeability tests were conducted (some of which were presented earlier) on oil sand fine tails to provide the necessary input parameters into the model. In order to verify the input parameters and the model, consolidation of MFT in a 10 m tall standpipe was modelled. The standpipe (and one other containing an MFT/sand mix) were erected at the U. of A. under the direction of Dr. J.D. Scott.

At the time of Pollock's work, the MFT had been consolidating for 4.5 years. The results of the modelling indicated very good agreement between the predicted surface settlement with time and the dissipation of excess pore pressures and the actual values. The solids content profile did not have as good agreement. Subsequent to the initial modelling, Suthaker and Scott (1994) published the standpipe data for an additional 5.5 years (*i.e.*, 10 years total). Again there was very good agreement between the predicted surface settlement with time and the dissipation of excess pore pressures and the actual values and less agreement for the solids content profile.

It is interesting to note that very little dissipation of excess pore pressure was observed in the 10 m standpipe after 10 years and that this is precisely what the Finite Strain theory predicts. That is, both observation and theory suggest that the hydraulic conductivity and compressibility of the MFT result in such small changes in excess pore pressure (after 10 years) that it is difficult to detect the changes with conventional pore pressure measuring equipment.

Several possible explanations have been offered to account for the solids content discrepancy including sampling difficulties, thixotropy, and creep. A non-destructive technique to measure the solids content may need to be employed.

5.6.2 Empirical Approaches

Several empirical approaches have been used to predict the behavior of MFT in the long term.

One method, presented by Devenney (1993), is to scale up laboratory bench tests by using a time relationship derived from Terzhagi's theory. That is:

$$\text{time to 90\% consolidation} \propto (\text{constant})(\text{drainage path})^2$$

where constant is back calculated from the laboratory tests.

Although, as discussed above, Terzaghi's theory is not applicable to slurries, this method provides some basis for which to extrapolate test results and obtain estimated consolidation times relatively easily. Pore pressure and solids content profiles are not available with this technique.

A second approach uses empirical data based on pond solids contents following from the work of Mackinnon and Sethi (1993). This work reports an empirical relationship between solids content and time. The empirical correlation is based on collecting solids content data from varying depths in a tailings pond. It is assumed that age is determined by depth.

It is reasonable to suggest that the empirical equations of the type developed by MacKinnon and Sethi (1993) can give a performance projection of likely future trends in the specific pond for which they were developed; but the application to more general predictions and the assertion that they confirm a creep mechanism is less certain. It is also reasonable to note that the Solids-Time empirical approach if extended to long time clearly suffers from the validity of the extrapolation from 10 or so years of data.

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VOLUME II

FINE TAILS AND PROCESS WATER RECLAMATION

CONTENTS

1.0 INTRODUCTION

The current commercial development of the Athabasca Oil Sands deposits in northeastern Alberta involves the Suncor Inc., Oil Sands Group at its Tar Island site, and the larger Syncrude Canada Ltd. plant, at its Mildred Lake site. The two plants produce an average of about 300,000 barrels (about 48,000 m^3) of synthetic crude oil per day, from the processing of about 600,000 tonnes of oil sands. Both facilities separate bitumen from oil sands based on a caustic hot water process. This extraction process uses large volumes of water (about 0.7 to 0.8 m^3 per tonne of oil sand) and as a result produces large volumes of fluid waste. This waste slurry, consisting of water, sand, clay, and unrecovered bitumen, is transported to a settling basin or tailings pond. Most of the coarser tailings sand quickly falls out and is used to build the dykes and beaches of these ponds. The fines fraction (silt/clay fraction) enters the pond and gradually develops, through densification, into the "mature" fine tails (about 30% solids by weight). A clarified surface layer of water also develops and is utilized as recycle water in the extraction plant.

A "zero discharge" policy restricts either operation from discharging the fine tails (or fine tails release water) to the surrounding environment. Consequently, a large inventory of fine tails is accumulating at a rate of about 0.1 to 0.2 m^3 per tonne of oil sands processed [1]. At the end of 1993, for instance, the tailings ponds of both Syncrude and Suncor contained a total of about 300,000,000 m^3 of fine tails. In fact, should processes continue as they have in the past, by the end of the current operations during the next century, volumes of fine tails approaching 1 billion m^3 could require reclamation.

Natural consolidation of the fine tails to a trafficable surface, capable of supporting a productive soil layer, would require hundreds of years (or more). The slow rate of consolidation of the existing fine tails is compounded by the continuous addition of new fine tails from the extraction process. Consequently, reclamation of the tailings ponds is considered to be the major environmental issue facing the surface mining oil sands industry.

Through regulatory approvals and corporate policy, Suncor and Syncrude operate under a commitment to reclaim their oil sands leases and accumulated waste materials in an environmentally acceptable manner. The proximity of the operations and the similarity of the extraction processes means a common reclamation research direction has evolved. This was accomplished through sharing of environmental research projects and their results, as well as wider efforts under the auspices of joint industry, government, private consultants, and university groups (*e.g.*, the Fine Tails Fundamentals Consortium).

Currently, no single reclamation option can handle the projected volumes of fine tails in a manner which is technically, environmentally, and economically acceptable. Consequently, the reclamation planning for handling of the fluid waste inventory requires an integrated approach. This means that the reclamation of the fine tails materials will be accomplished through a combination of the so called "dry" and "wet" landscape techniques. The dry approach involves dewatering or incorporation of fine tails into a solid deposit which is capable of being reclaimed as a land surface, or a wetland. The wet approach involves a lake system, whereby contained fluid fine tails are capped with a layer of water of sufficient depth to isolate the fine tails from direct contact with the surrounding environment. A component of both approaches would likely include the use of wetlands, to act as either a buffer between reclamation units and the receiving environment, or as an active bioreactor treatment system for the retention and degradation of potentially deleterious components. Each approach has advantages and disadvantages, thus the final strategy will depend on detailed technical and environmental assessments, consideration of economic reality, and stakeholder acceptance. These assessments will also be incorporated within an overall reclamation risk evaluation. Regardless of the actual techniques employed, the reclamation objective is to produce an area that is stable (good erosion control and geotechnical stability), productive (for both terrestrial and aquatic biota, with no long term toxic effects), and self-sustaining (diverse ecosystem development, maintenance-free).

1.1 Background

Initial characterization and toxicity testing of the oil sands tailings waste streams began in 1975 on the Great Canadian Oil Sands lease (now Suncor). Tailings recycle water had a high organic carbon content, relative to the Athabasca River, and was acutely toxic to rainbow trout (LC_{50} <20%). Preliminary characterization of the organic fraction by gas chromatography (GC) could not pinpoint the causes of the toxicity. Following these initial assessments a number of studies were initiated, involving more detailed characterization of the organic content of various wastewater streams.

Chemical characterization of the tailings settling basin reveals dissolved organic carbon (DOC) concentrations in the range of 100 mg carbon/L. Most of the dissolved organics in the tailings (>80%) are organic acids, including surfactants, phenols, and alcohols. Quantitatively, only a minor amount (<5%) of the organic matter is in the base/neutral fraction. A more detailed examination of the base/neutral fraction shows that most are aliphatic and polar hydrocarbons [2]. Aromatics (such as the Polycyclic Aromatic Hydrocarbons (PAHs) and other Priority pollutants) are present in small amounts.

The water associated with freshly produced fine tails is acutely toxic to aquatic organisms at various trophic levels (from bioassay tests of bacteria, *Daphnia*, fathead minnows, and trout). The same toxic effect is observed in bioassays of the water contained within both the Syncrude and Suncor tailings ponds, as well as laboratory generated extraction tailings waters.

For the purposes of this work, toxicity is defined as that property of a material which results in an adverse effect in a living organism. More specifically, there are degrees or levels for expressing toxicity, which can be defined as either *acute toxicity* (a relatively fast and distinct measurable response, such as reduced light emission or death), or *chronic toxicity* (a measurable response over a relatively long period of time, such as changes in growth rate, maturation, flesh tainting, or carcinogenic effects.

Essentially all of the acute toxicity of the oil sands fine tails has been found to be associated with the acid extractable organic fraction of the tailings water. Through comparison with infrared spectra of standards, the predominant organic component found within this fraction is a complex mixture of carboxylic acids, known as naphthenic acids. There is no doubt as to the presence of these naphthenic acids, which are natural surfactants found in the alkaline extract of crude oils [3]. These compounds are present in the bitumen of the oil sands and are released during the hot water extraction process. They play a key role in the bitumen extraction process itself [4], and are concentrated because the tailings pond water is recycled many times.

1.2 Reclamation Options

The wet and dry landscape options can be defined in general terms, with each having similar environmental requirements for the initial reclamation phases. Each fine tails management and reclamation program must ensure that:

(a) there is restriction of direct contact or release of contaminants into the environment;

(b) there is restriction of off-site transport of contaminants, which can be mobilized by seepage;

(c) there is restriction of hydrological impacts on the fine tails deposits;

(d) the landscape is stable;

(e) the landscape is productive; and

(f) the reclaimed area is self-sustaining.

1.2.1 Dry Landscape Options

Dry landscape techniques aim to significantly reduce the water content of fine tails and potentially produce a solid deposit which would be capable of being capped with soil as part of terrestrial (or wetlands) reclamation scenario [5–9]. Some of the various dry landscape options include:

(a) dewatering of the fine tails to a more fully consolidated clay through processes such as evaporation and freeze-thaw;

(b) incorporation of fine tails with overburden clays (adsorption of fine tails pore water with clay mixes) and with coarse tailings sand (enhanced capture of fine tails solids in the coarse tailings deposit); and

(c) chemical alteration of clay components in tailings prior to deposition (*e.g.*, lime, acid/lime, and calcium sulphate addition to extraction tailings to produce a non-segregating or consolidated tailings).

In all of these dry landscape approaches, the fluid character of the fine tails is reduced significantly, and in some cases, the produced materials will be strong enough to allow for soil capping and terrestrial reclamation. Inherent within all dry landscape options is the requirement to deal with waters that may drain or leach from the reclamation areas because these waters may contain unacceptable levels of organic acids or dissolved ions.

1.2.2 Wet Landscape Options

The wet landscape option leaves the fine tails as a fluid, which means it remains non-trafficable and requires containment in geotechnically secure areas. Containment areas are available since the mining operation results in mined-out pits. As long as these pits are geotechnically stable, they can be used for long-term secure storage of fluid materials.

Conceptually, the wet landscape method disposes of mature fine tails as a fluid, over which a layer of water is placed [10,11]. The depth of the water cap layer is optimized to ensure both the isolation of the fine tails zone from physical mixing with the overlying water layer, and the establishment of a viable, self-sustaining aquatic ecosystem in the climatic conditions of northern Alberta.

This water capped lake is expected to form a productive system, with the detritus-covered fine tails zone forming the lake sediments. This detrital layer will act both as a barrier for potential mixing from the capping layer, as well as a biologically active zone for the degradation of organic acids from the fine tails layer.

The principle upon which the water capping reclamation approach is based is that the rheological properties of mature fine tails (low permeability, high density, relatively high yield-strength and viscosity) are conducive to the success of the capping scheme. As a result of these properties, the establishment of a well-stratified aquatic system is possible, with two distinct layers and very low potential for active mixing of the zones [12]. Gradual release of the fine tails pore water into the overlying water cap will occur as the fine tails densifies. However, the release rates will be slow due to the low consolidation rates of the fine tails material. Natural detoxification processes occurring in the water cap layer and in the detrital layer will degrade or mineralize any potentially negative components mobilized with the released pore water.

1.3 Wetlands

Some water release will occur from both the wet and dry landscapes. Dry landscape areas will have seepage of entrained porewaters, groundwater discharge as part of the hydrological cycle, or runoff/drainage from the reclaimed areas. Wet landscape areas will have a water discharge from the lake system because of inflows to the lake of drainage waters from the surrounding land surfaces, as well as precipitation and groundwater discharges. Both reclamation approaches include wetlands as an integral component of the reclamation process [13]. While the quality of waters from the fine tails reclamation areas is expected to be high, the wetlands will function as a safeguard or buffer zone between the reclaimed deposits and any off-lease receiving aquatic environment.

Two types of wetlands will be associated with fine tails reclamation: those constructed as receiving areas for waters draining from either the dry or wet landscape options, and those which develop within the shoreline littoral zone of the water capped fine tails lakes. As such, these wetland systems would equate to either constructed wetlands in the first case, and to naturally developing systems in the second case. These wetlands will be shallow and highly productive. They will allow for the biodegradation or adsorption of components in the water escaping from reclamation landscapes.

To summarize, the objective of this volume on "Fine Tails and Process Water Reclamation" will be to highlight research results that are crucial to either the wet or dry landscape strategies. Specifically, this research involved: (1) evaluating the concerns of acute and chronic toxicity, (2) determining leachate potential of reclamation material, (3) development of specific design criteria for wetlands, and (4) development of an ecosystem model to assist in defining the overall fine tails reclamation strategy.

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2.0 QUALITY OF FINE TAILS

2.1 Sources and Pathways of Chemical Constituents

2.1.1 Parent Materials

Most of the waste materials that comprise the tailings associated with oil sands processing are derived from the oil sands themselves [1]. The oil sands come from the McMurray Formation, a deltaic sand deposit in a transgressive Cretaceous structure [2]. This unit tends to be coarser grained at the bottom and finer grained toward the top [3]. The bitumen is present in the intergranular pores of sands and fine-grained micaceous sands, leading to complex variations in the degrees of bitumen saturation over three dimensions.

Oil sands can vary in grade, fines or clay content, mineralogy and salt content [1]. Typical oil sands consist of approximately 10 wt% bitumen; the remainder is made up of roughly 85% coarse sand (>22 μm) and a 15% fines (<22 μm) fraction, consisting of silts and clays [4].

Organic compounds are the primary contaminants associated with oil sands wastes, and bitumen is the major source of the organic compounds. Bitumen generally refers to the solid members of petroleum [5]. Bitumen is an extremely complex mixture of organic compounds, with a typical range of molecular weights from 490–620 g/mol [5]. Carbon accounts for about 80–85% of the elemental composition of bitumen, hydrogen ~10%, sulphur ~5%, and nitrogen, oxygen, and trace elements the remainder [5,6].

The primary source for inorganic contaminants (*e.g.*, salts and metals) associated with the oil sands are the connate waters and clay minerals.

2.1.2 Tailings Sand

Tailings sand is one of the by-products of bitumen extraction. Bitumen is separated from the ore using the Clark Hot Water Extraction process, which involves dispersion of the oil sands with water, steam, mechanical conditioning, and possible addition of caustic (NaOH), with the subsequent separation of bitumen from the sands. A significant by-product of the extraction process is the tailings stream, which contains water, unrecovered bitumen, a coarse sand fraction (>22 μm), and a fines fraction (<22 μm). The tailings stream is discharged into tailings/settling ponds, where the coarse solids segregate from the fines and are used to build the dykes that surround the tailing/settling ponds (Figure 2-1).

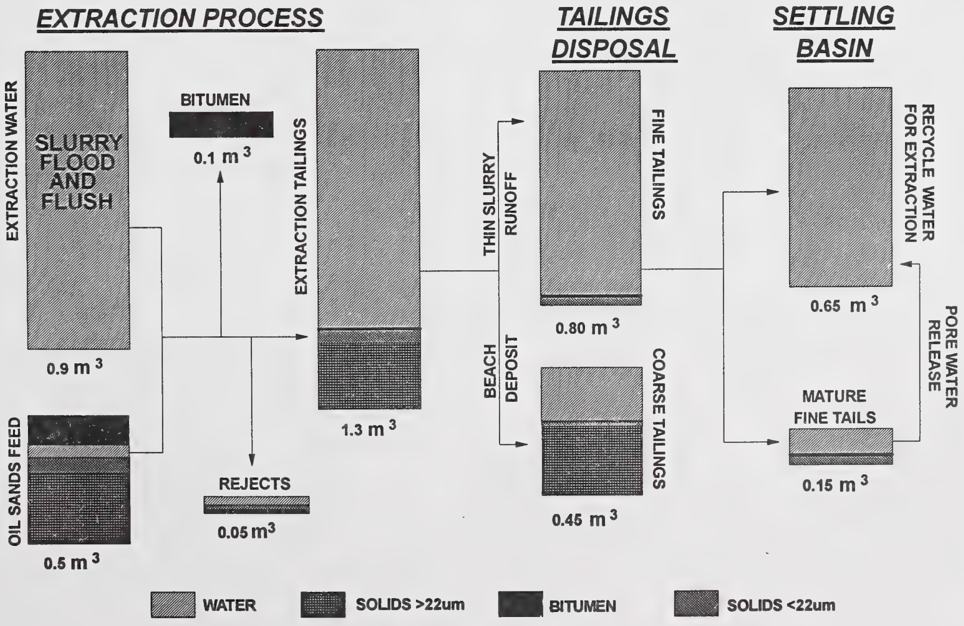


FIGURE 2-1. Oil sands extraction and waste disposal process [4].

This coarse fraction consists primarily of sand particles, with small amounts of unrecovered bitumen entrained in the sand. The sand is 95.5 – 99% SiO_2, 0.5 – 0.9% Al_2O_3, and 0.1 – 0.9% Fe [4]. While the sand itself is essentially inert, the unrecovered bitumen within the sand dykes is a potential source of contaminants to the environment as a result of dissolution into groundwater.

2.1.3 Fines Fraction

As noted above, fine tails are a by-product of the extraction process, during which the fine mineral grains become desegregated and dispersed [7]. These fines run off into the tailings/settling pond as a thin slurry and become entrained in the surface water layer where they remain suspended for periods from several hours to up to three months. Following this induction period, the fine particles settle into the fine tails layer of the pond (Figure 2-1).

Chemical and toxicological properties of both the fine tails and porewater (*i.e.*, water within the void spaces of the fine particles) are a function of the source of the ore. Properties of oil sands change with location as a result of differences in origin, facies, and depositional and post-depositional environments. For example, the <44 μm fraction can vary from less than 5% to greater than 40% of the tailings solids, depending upon the source of the ore. This fraction has averaged 20 – 22% at Syncrude but only 11 – 13% at Suncor.

The main clays identified in the less than 2 μm (clay) fraction from Syncrude's settling basin are the layer silicates (*e.g.*, kaolinite, hydrous micas such as illite and muscovite, and chlorites) and quartz [1]. In the upper levels of the fine tails zone, clay minerals predominate while the quartz content of the solids increases with depth into the fine tails [8].

Residual bitumen accounts for about 1.5 – 2.0% of the fine tails of Syncrude's settling basin, while at Suncor's ponds it ranges from 2 – 3% in Pond 3 to 6% in Pond 1. The content of light hydrocarbons (unrecovered diluent such as naphtha) is about one-tenth of the bitumen content [1]. The hydrocarbons contained in the fine tails constitutes a large pool of contaminants that requires effective control to prevent or manage release to the environment.

2.1.4 Liquids

Considerable amounts of water are required for bitumen recovery, hence large volumes of fluid wastes are produced. Water associated with the transport of the tailings slurry to the tailings/settling ponds forms the "free water" zone of the ponds (suspended solids <0.1% at Syncrude and <1% at Suncor). Water in the free water zone is recycled and no extraction process waters are released from the sites to the surrounding environment [1].

Dewatering of the fine tails, which results in expression of the porewater into the free water, occurs rapidly for about two to three years, proceeding to a solids content of ~30%. Further dewatering of these mature fine tails (MFT) occurs extremely slowly, with hundreds of years possibly being required to develop properties associated with soft soils [7].

Most of the constituents associated with fine tails water are derived from leaching of the oil sands [4]. The highest grade oil sands contains 2 – 6 wt% (10 – 15% pore volume) connate water, but as ore grade decreases connate water increases [9]. In addition, both make-up water taken from the Athabasca River for use in plant processes, as well as chemicals added during hot water extraction and other processes, add inorganic and organic compounds to the water [4]. A small fraction of water soluble organics are also released during the hot water extraction process. These soluble organics are primarily carboxylic and sulphonic surfactants, which are components of bitumen.

2.2 Chemical Characterization

2.2.1 Inorganics

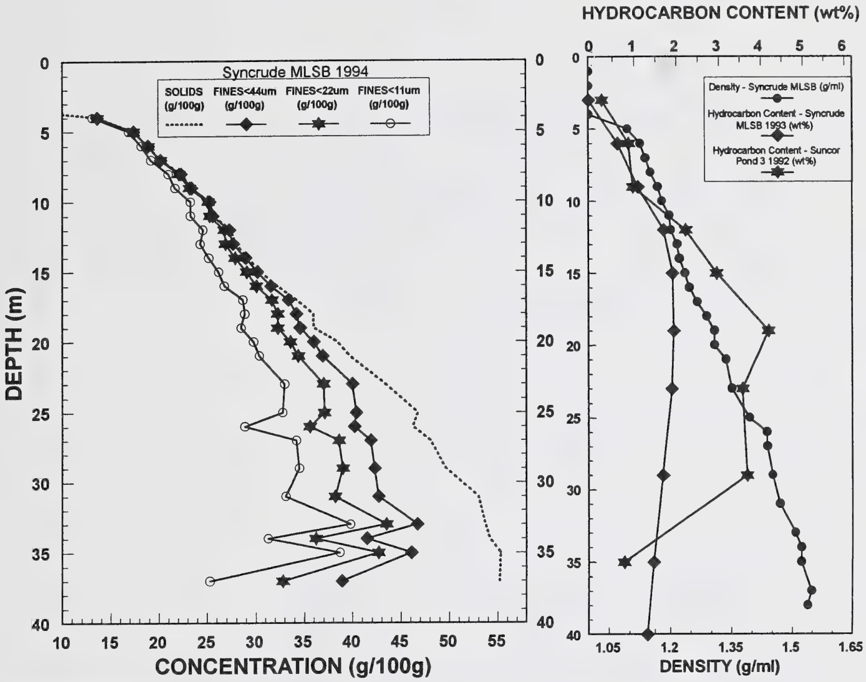
2.2.1.1 *Fine Tails*

There are changes in particle size, mineralogy, and elemental composition of the MFT with depth in the tailings/settling ponds. Both density and particle size increase with depth in all ponds as total solids content increases (Figure 2-2). These changes are a result of segregation and settling of the coarser sand particles through the fine tails [4].

There is a shift in the elemental composition of the solids fraction, in addition to changes of particle size and mineralogy with depth in the fine tails (Figure 2-3). Both Al and K content of the fine tails solids decrease with depth, whereas Si content increases with depth. These trends are consistent with that expected based on changes in the particle size and mineral distribution as a result of quartz sand accumulation within the fine tails [1].

Total sulphur content of fine tails varies from about 0.5% in Syncrude's settling basin to 1.0% in Suncor's tailings ponds [10]. About 60 – 70% of this sulphur was organic and the remainder sulphide.

The metals and trace elements detected in fine tails are consistent with those detected in oil sands samples (Table 2-1). Metal concentrations in the fine tails samples are, however, generally elevated compared to concentrations in the oil sands. This is not surprising since most metals are associated with the fine clay minerals, which comprise the bulk of the fine tails, rather than the coarser sands.



FINES=FINES/(FINES+WATER)

FIGURE 2-2. Distribution of density, particle size, solids, and bitumen levels with depth in MLSB [8].

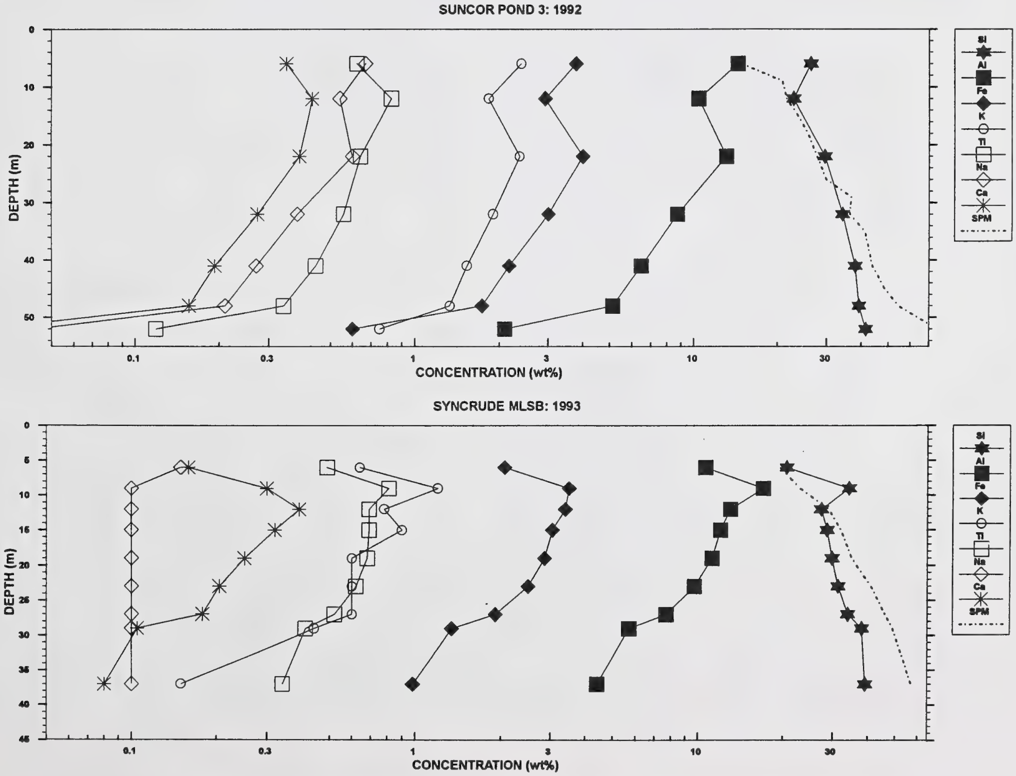


FIGURE 2-3. Elemental content of solids in Syncrude's MLSB and Suncor's Pond 3 [4].

TABLE 2-1. Metals content of oil sands, fine tailings, and process water

Element	Solids (ug/g)				Water (mg/L)		
	Syncrude oil sands	Suncor oil sands	Syncrude MLSB fine tailings	Suncor Pond 2 fine tailings	Syncrude MLSB "free water zone"	Syncrude MLSB fine tailing zone (16 m)	CCREM guidelines for freshwater aquatic life
Aluminum	789	748	3440	2690	0.1	0.76	0.1
Antimony	<0.05	<0.05	<0.05	0.06	0.0012	0.0028	–
Arsenic	1.75	1.55	5.54	5.35	0.0072	0.0340	0.05
Barium	16.9	18.7	77.9	59.7	0.10	0.09	–
Beryllium	0.4	0.4	1.1	1.5	<0.001	<0.001	–
Boron	–	–	–	–	2.75	2.74	–
Cadmium	<0.3	<0.3	<0.3	<0.3	<0.003	<0.003	0.0013
Calcium	569	2570	2400	3340	–	–	–
Chromium	5.6	2.0	15.0	6.8	<0.002	0.004	0.02
Chromium (hexavalent)	–	–	–	–	<0.003	<0.003	0.003
Cobalt	4	4	11	12	<0.003	0.008	–
Copper	3.4	2.0	8.5	13.5	0.005	<0.001	0.003
Iron	4320	7450	13800	18100	0.01	0.18	0.3
Lead	<2	<2	7	9	<0.02	<0.02	0.004
Lithium	–	–	–	–	0.134	0.113	–
Magnesium	424	1230	1730	1680	–	–	–
Manganese	153	217	472	518	0.026	0.008	–
Mercury	–	0.02	0.05	0.04	<0.00005	<0.00005	0.0001
Molybdenum	<2	<2	<2	<2	0.183	0.045	–
Nickel	16	15	23	39	0.014	0.014	0.11
Phosphorus	47	54	162	149	–	–	–
Potassium	322	267	1220	858	–	–	–
Selenium	<0.02	<0.02	0.09	0.10	<0.04	<0.04	0.001
Silicon	–	–	–	–	2.0	4.4	–
Silver	–	–	–	–	<0.002	<0.002	0.001
Sodium	257	207	1800	1370	–	–	–
Strontium	–	–	–	–	0.254	0.193	–
Titanium	–	–	–	–	<0.003	0.003	–
Uranium	–	–	–	–	<0.5	<0.5	–
Vanadium	22.5	26.2	28.9	75.1	0.003	0.021	–
Zinc	12.8	10.7	37.5	41.2	0.010	0.012	0.03

2.2.1.2 *Porewater and Tailings Pond Water*

The primary source of dissolved components in fine tails porewater is the release of leachable material from the oil sands during bitumen extraction. Other sources, such as the process chemicals and components in water imports are relatively minor. Table 2-2 summarizes the composition of water with depth at Syncrude's settling basin and Suncor's Ponds 1, 2, and 3. The characteristics of the wastewaters in the tailings/settling ponds show many similarities and a few differences, including:

- pH ranges from 7.6 – 8.4, is comparable in all ponds, does not vary greatly with depth, and is within the range observed in natural surface waters from the region.
- Conductivity levels are about twice as high in the free-water zone of Syncrude's settling basin (2400 μS/cm) compared to Suncor's ponds (~1100 μS/cm). However, levels in the fine tails porewater are slightly higher in Suncor's ponds (1400 – 2000 μS/cm) compared to Syncrude's settling basin (1250 – 1500 μS/cm). These trends may reflect differences in ionic composition of the ore, changes in ionic content of the processed oil sands, and *in situ* changes that might affect the ionic concentration and composition of pore waters [1].
- Ammonia concentrations are generally high in wastewaters from both plants, resulting in concentrations within the free-water zone of about 3.5 mg/L in Syncrude's settling basin and 4.7 – 6.6 mg/L in Suncor's ponds. Concentrations in Suncor's fine tails porewater range from 4 – 51 mg/L and are considerably higher than in Syncrude's settling basin (3.3 – 7.1 mg/L). These differences likely reflect input of upgrading area wastewaters to the tailings stream at Suncor, which contains high ammonia concentrations.
- Water from Syncrude and Suncor ponds differs in both the absolute and relative concentrations of major ions. For instance, chloride concentrations from Syncrude's settling basin are as much as 10-fold higher than from Suncor's tailings ponds, while the reverse trend has been observed for sulphate (the elevated sulphate levels reported for Suncor may, however, be an artifact of sample storage [1]). In addition, although sodium is the dominant cation in all ponds, concentrations in Syncrude's settling basin are higher than in Suncor's ponds. The differences in major ion composition reflect differences in the quality of the ore and the use of caustic (sodium hydroxide), *i.e.*, Syncrude uses about three times more caustic during extraction than Suncor.
- Boron is a significant component of the leachates from oil sands and its concentration is relatively constant with depth in the tailings/settling ponds [8].
- Trace metal concentrations in fine tails porewater are generally low relative to regulatory guidelines for protection of freshwater aquatic biota (Table 2-1).

2.2.2 Organics

2.2.2.1 *Fine Tails*

Several broad classes of the organic components associated with fine tails have been studied. These classes have been defined primarily by how they have been isolated and secondarily on their chemical and structural behavior. The major classes include (1) residual bitumen or toluene-soluble fraction, (2) strongly bound organic matter (SOM) extracted using supercritical methanol, (3) humic substances extracted with strong base, and (4) aqueous-phase dissolved organics of varying volatility [11].

The major hydrocarbon components of fine tails include unrecovered bitumen and naphtha from the diluent recovery system. Bitumen content of the fine tails is about 1 – 2% in Syncrude's settling basin and 1 – 9% in Suncor's ponds [1]. The hydrocarbon concentrations show more variability and depth dependence than the mineral solids (Figure 2-2).

Levels of non-polar organic compounds such as polycylic aromatic hydrocarbons (PAHs) and heterocyclic aromatic hydrocarbons (*e.g.*, thiophenes) in the fine tails are generally in the range of <0.01 – 10 mg/kg (Table 2-3). The highest concentrations are generally associated with alkylated forms rather than the parent compounds.

The ease of bitumen recovery from oil sands depends upon the surface properties of the components, particularly the water wet characteristics of the clay and sand particles [12]. Oil wetting of some of the oil sands solids is caused by the presence of SOM. In addition, it is believed that the presence of SOM is partly responsible for the intractable behavior of the fine tails after hot water extraction of oil sands [12]. A combination of alkaline and supercritical gas extractions was used to characterize the SOM. The dominant groups identified included: aliphatic organics, methoxy and ethoxy carbons, unsubstituted and bridgehead aromatic carbons, and carboxylic acids. The humic acid portion of the organic rich solids can be separated by alkali treatment [12]. Humic acids associated with the fine tails are predominantly composed of aromatic hydrocarbons.

2.2.2.2 *Porewater and Tailings Pond Water*

The levels of dissolved organic carbon (DOC) reported in both the surface and pore waters from the settling ponds are fairly low (55 – 85 mg/L), considering the high oil content in the extraction stage and the potential for input of organics by

TABLE 2-2. Comparison of the chemical properties of Syncrude and Suncor's tailings ponds for 1992 [1]

Depth (m)	Temp (°C)	pH	Cond (μS/cm)	Solids (g/100 g)	Bitumen (g/100 g)	Microtox		Major ions (mg/L)							Nutrients/metals (mg/L)						
						IC_{50} (%)	IC_{20} (%)	Na	K	Mg	Ca	Cl	SO_4	HCO_3	DOC	NH_3	PO4	Fe	Si	Al	B
Syncrude																					
3	20	8.0	2400	0.025		31	9	550	9.2	3.1	4.6	220	210	851	58	3.5	0.005	0.08	1.75	0.07	2.1
5.5				18.40	0.7																
7	12	8.0	1500	21.30	0.8	29	8	510	9.9	2.7	6.8	160	7	900	58	3.3	0.011	0.08	3	0.3	2.5
12	13	8.2	1325	27.80	1.6	42	10	480	12.3	2.9	5	135	6	940	63	7.1	0.077	0.17	3.6	0.44	1.9
17	18	8.2	1250	35.70	2.0	23	7	480	12.2	2.7	4.3	135	3	960	66	6.2	0.142	0.13	3.5	0.43	2.3
24	20	8.2	1250	41.50	1.8	32	8	480	12.2	2.7	3.7	125	1	968	77	6.3	0.186	0.27	4.05	0.8	2.4
30	13	8.3		49.50	1.7	27	8	475	14.0	3.2	4.4	126	12	995	75	6.8	0.005	0.34	4.22	1.21	2.4
Suncor Pond 1																					
1		7.8	1113	0.60		51	10	366	18.1	11	19	43	72	813	62	6.6	0.005				
3	15	8.0	1508	23.10	5.0	61	12	373	18.9	9.6	19	27	9	991	65	14	0.005	0.62	9.14	2.52	3.8
6	15	8.0	1853	38.30	9.1			380	25.6	22	17	18	49	1271	68			0.27	8.37		
9	18	7.9	2080	37.40	7.7	57	13	418	28.5	25	21	14	39	1474	65	51	0.041	0.12	8.04	0	3.8
12	20	8.0	1939	40.80	5.6	94	20	399	28.3	24	17	9	58	1332	58			0.24	8.16		
14	20	8.2	1625	44.20	9.9			358	22.5	17	16	10	73	1042	75			0.63	10.1		
16	21	8.4	1382	48.70	7.8	100	13	309	21.8	14	13	12	15	905	78	22.7	0.08	0.88	11.1	3.71	2.4
Suncor Pond 2																					
1		7.8	1160	2.20		61	10	384	8.4	5	24	45	87	859	65	4.7	0.006				
4	15	8.1	1385	24.80	2.0	64	12	376	19.8	5.8	6.6	27	42	890	70	10	0.005	5.79	48.6	26.8	4.4
6	16	8.2	1521	27.90	5.0			387	19.9	6.1	9.3	22	108	890	67			1.93	19		
9	19	8.2	1470	31.00	4.9	80	16	368	25.7	8.5	11	21	84	890	69			6.06	48.1		
16	21	8.2	1379	40.50	3.7			358	16.2	4.2	5.5	25	31	874	60	14.5	0.005	1.59	16.7	6.9	4.3
19	22	8.2	1427	45.70	4.0	83	15	371	14.5	4.8	6.7	26	47	874	60			1.17	13.1		
22	21	8.3	1383	51.80	2.7			372	15.2	4.5	6.5	25	62	890	58			0.97	12.8		
28	20	8.3	1421	43.10		91	17	384	15.4	5.5	7.7	26	44	890	63	14.2	0.005	1.19	12.8	5.1	3.9
Suncor Pond 3																					
1		7.7	1128	1.20		45	8	382	10.0	18	27	47	82	859	67						
6	20	8.2	1487	14.80	0.3	78	15	422	14.8	5.2	8.3	34	81	874	69	6.6	0.005	2.31	17.7	8.38	3.2
8		8.0	1690			52	11					40	7	935	64						
9	16	7.9	1448	20.80	0.9			423	15.1	4	6.2	34	42	915	73			3.84	31.3		
12	18	8.0	1453	21.80	1.0	64	13	430	15.2	4.5	6.4	29	57	905	73	3.9	16.2	3.6			
15	18	7.9	1397	23.80	2.2			400	12.2	3.3	5.4	27	35	905	68	9.2	0.005	1.25	13.5	5.05	3.7
18	18	8.0	1410	25.80	3.2			404	11.5	3.4	5.4	28	43	874	68			1.21	14		
22	19	8.2	1434	27.70	2.9	77	19	419	12.9	3.9	5.9	27	35	905	65			1.67	17.3		
28	19	8.0	1459	29.90	2.0			414	12.8	4.3	7.1	26	82	864	64	10.9	0.005	0.7	10.9	2.42	3.9
29	19	8.2	1411	36.70	4.1			399	11.6	3.6	5.8	26	56	874	63			0.84	11.6		
32	19	8.1	1399	36.40	4.7			398	12.0	3.7	5.7	27	45	874	63			0.76	10.9		
35	19	8.1	1423	41.20	3.5			398	12.4	4	6.2	26	71	839	63	10.3	0.007	0.98	12.4	3.83	3.7
38	20	8.0	1505	38.50	2.6	76	15	433	13.1	4.8	8.1	28	139	864	64			0.8	11.2		
41	19	8.0	1474	43.80	3.6			419	12.2	4.1	6.5	28	71	890	60			0.59	9.78		
45	19	8.0	1500	48.80	2.1			424	12.6	4.3	6.8	27	93	890	64	9.7	0.013	1.06	11.3	2.98	3.7
48	18	8.1	1637	54.40	0.8	100	27	450	14.5	6	9.7	29	179	839	65			0.39	7.98		
52	18	8.1	1533	75.70	0.5			432	20.4	6.8	7.7	32	137	813	83	8.7	0.005	8.3	77.9	46.8	3.9

TABLE 2-3. Polycyclic aromatic hydrocarbons (PAHs) in solids and leachates

	Solids (g/g)				Leachate (g/L)	
	Syncrude oil sands	Suncor oil sands	Syncrude MLSB fine tailings	Suncor Pond 2 fine tailings	Syncrude oil sands	Suncor oil sands
Parent compound PAHs[1]						
Range[3]	<0.01–4.9	<0.01–7.0	<0.01–2.6	<0.01–3.3	<0.04–0.23	<0.04–0.21
Total[4]	13.15	16.55	8.53	10.45	0.41	0.40
Alkyl substituted PAHs[2]						
Range[3]	<0.01–36	<0.01–55	<0.01–9.7	<0.01–50	<0.04–0.33	<0.04–0.38
Total[4]	190.4	296.75	54.67	228.28	0.33	0.38

[1] Parents compound PAHs include: naphthalene, acenaphthylene, acenaphthene, fluorene, dibenzothiophene, phenanthrene, anthracene, fluoranthene, pyrene, benzo(a)anthracene, chrysene, benzo(b&k)fluoranthene, benzo(a)pyrene, indeno(c,d-123)pyrene, dibenzo(a,h)anthracene, and, benzo(g,h,i)perylene.

[2] Alkyl-substituted PAHs include: methyl naphthalene, C_2 substituted naphthalenes, C_3 substituted naphthalenes, C_4 substituted naphthalenes, biphenyl, methyl biphenyl, methyl acenaphthene, methyl fluorene, C_2 substituted fluorene, methyl phenanthrene/anthracene, C_2 substituted phenanthrene/anthracene, C_3 substituted phenanthrene/anthracene, C_4 substituted phenanthrene/anthracene, 1-methyl-7-isopropyl-phenanthrene (retene), methyl dibenzothiophene, C_2 substituted dibenzothiophene, C_3 substituted dibenzothiophene, C_4 substituted dibenzothiophene, methyl fluoranthene/pyrene, methyl benzo-(b&k)fluoranthene/methyl benzo(a)pyrene, and C_2 substituted benzo(b&k)fluoranthene/benzo(a)pyrene.

[3] Range of concentrations reported for individual compounds.

[4] Sum of concentrations reported for all compounds.

on-site activities. Samples analyzed from Suncor's tailings ponds indicate that most of the DOC is contained in the <1000 MW fraction, which would include naphthenic acids as well as more volatile organic acids and phenols. In addition, the samples contained a small, but measurable amount of high molecular weight organics (5000–30000 MW).

Acid-extractable organics account for most of the acute toxicity in tailings pond recycle water and about 80% of toxicity in fine tails porewater from both Syncrude's and Suncor's ponds [13,14]. Up to 95% of the total acid fraction extractable from fine tails is composed of naphthenic acids, and these compounds are associated with most or all of the acute toxicity expressed by tailings pond water or fine tails porewater [14].

Naphthenic acids are predominantly carboxylic acids with a cyclic or polycylic alkane backbone (cyclopentene and cyclohexane) and aliphatic side chains of various lengths [15,16]. Herman *et al.* present a general structural formula for naphthenic acids (Figure 2-4A) [16]. In this structure, *m* has a value of two or more, and *R* is a small methyl or ethyl group. Conversely, the short chain could be the –COOH group inserted directly on the carbon ring, and the long chain of *m* length could be the aliphatic side chain (Figure 2-4B). The number of cycloalkane rings normally varies from two to six. Both five-member and six-member carbon rings, or combinations of the two, may be present in naphthenic acids. Figure 2-4C, from [15], presents structures of typical naphthenic acids of various sizes using the second formula (*R* in these diagrams refers to the aliphatic side chain).

The range of molecular weights for naphthenic acids in crude oil ranges from 200 to >700, with a peak near 300 [15], but the heavier acids, with MW>450, are evidently a minor fraction in bitumen tailings [17]. Zenon reports that most carboxylic acids in tailings ponds have carbon chains of C_8 to C_{30}, indicative of molecular weights in the range 275–450 [13].

Naphthenic acids act as surfactants (compounds which modify the strength of surface attraction), and have both hydrophilic character as a result of the polar carboxyl group and hydrophobic character due to the nonpolar aliphatic end [14]. While naphthenic acids are a natural component of bitumen, they are liberated from the bitumen during the extraction process [18]. The addition of caustic soda accelerates the liberation of naphthenic acids into the process waters, where the acids are present as sodium salts (sodium naphthenates), rather than the parent acid, and the concentration of naphthenic acid in the wastewater slurry is correlated to the amount of sodium hydroxide added during extraction (Figure 2-5).

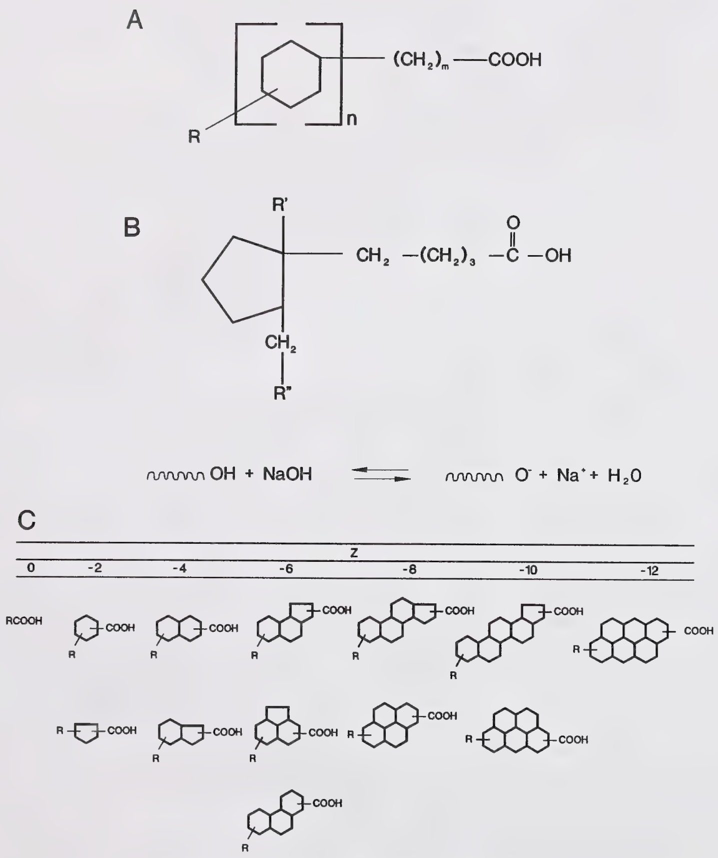


FIGURE 2-4. Naphthenic acid structure. A. after [16]; B. after [22]; C. after [15].

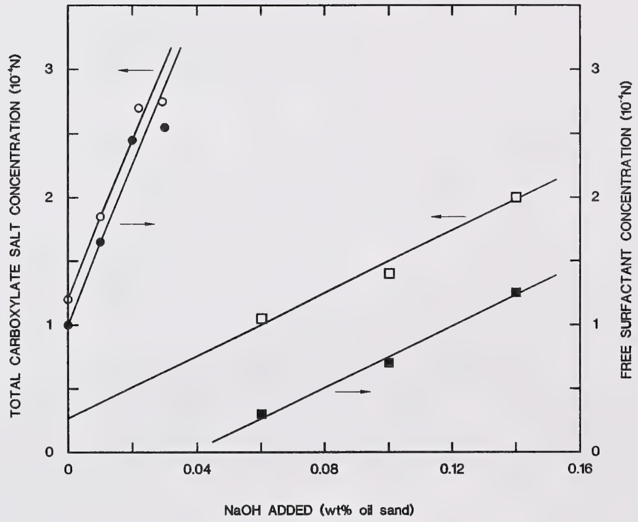


FIGURE 2-5. Concentrations of total carboxylate salts (open symbols) and carboxylate surfactants (filled symbols) *versus* NaOH addition. Effects on both rich (circles) and lean (squares) oil sands are shown. After [22].

The polarity and increasing non-volatility with increasing molecular weight make naphthenic acids difficult to measure by conventional mass spectrometry techniques [19]. Two different techniques are being used to analyze naphthenic acids — Fast Atom Bombardment Mass Spectrometry (FABMS) and Fourier Transform Infrared Absorbance (FTIR). FABMS provides a qualitative analysis of naphthenic acids by detecting the presence of a variety of naphthenic acids, ranging from open chain to one, two, and three ring moieties in their structure [19]. Semi-quantification has also been achieved with this method by calculating the apparent relative concentrations for the different *Z* families.

Syncrude Canada Ltd. is presently developing a method that appears to be promising for quantifying total naphthenic acid concentrations using FTIR and absorbance at two wave numbers present in the 1700 – 1800 cm^{-1} range. Typical concentrations of compounds from the naphthenic acids group, based on the FTIR method, range from 1 – 2 mg/L in the Athabasca River to over 100 mg/L in fresh tailings water (Table 2-4).

TABLE 2-4. Naphthenic acid concentrations (FTIR method)

Water source	Concentration mg/L
MLSB	112
Suncor Pond 1/1A	82
Suncor dyke drainage	41
Athabasca River porewater	
1. Along Tar Island Dyke	6.0
2. Upstream Tar Island Dyke	8.5
Athabasca River	1.9

In freshly collected fine tails, up to 15% of the acute toxicity of Syncrude's porewater and 35% of the toxicity of Suncor's porewater is associated with volatile organic compounds. These light-end hydrocarbons enter the tailings ponds as part of the slurry, with some lost to the atmosphere *via* volatilization and the remainder trapped in the fine tails [1]. The concentrations of the lighter hydrocarbons range from about 0.01 – 2%, depending upon the pond. BTEX compounds totalled about 0.1 mg/L in a sample of porewater squeezed from Suncor's Pond 1A fine tails [20].

Hargesheimer *et al.* characterized simple phenols from a total of nine water samples collected from various depths of Syncrude's settling basin, process-affected water associated with dyke drainage, and natural groundwater and surface waters from near the settling basin [21]. Concentrations of phenols and cresols ranged from 25 – 152 μg/L in samples from the settling basin, with concentrations decreasing sharply with depth into the fine tails. A number of simple alkylphenols were also identified in the settling basin samples. The decrease in concentration with depth may reflect adsorption of these compounds to particulate matter. Samples from dyke drainage, groundwaters and surface waters contained <1 μg/L of the simple phenols analyzed and did not contain any of the simple alkylphenols identified in the settling basin samples. A sample of surface water that drained over exposed oil sands contained low concentrations of phenol (4 μg/L), but no detectable concentrations of cresols or simple alkylphenols.

Concentrations of other organic compounds, such as PAHs are generally less than 0.01 μg/L in fine tails porewater, although values of up to 0.4 μg/L have been measured (Table 2-3).

2.3 Toxicity

Fine tails and the water associated with freshly produced fine tails are toxic to aquatic biota. Toxicity is defined as that property of a material that results in an adverse effect in a living organism. This effect can range from the death of the test organism to the inhibition of some quantifiable biological function such as change in growth rate [20]. Moreover, there are different levels for expressing toxicity:

- *Acute toxicity* — a distinct measurable response (*e.g.*, light emission, maturation, death) recorded after short-term exposures (*e.g.*, minutes to days) relative to the life span of the test organism.
- *Chronic toxicity* — a measurable response (*e.g.*, behavioral, physiological, reproductive, genetic) in the test organism or population recorded after long-term exposures (*e.g.*, days to weeks) relative to the life span of the test organism.

Considerable effort has been expended on evaluating the toxicity of oil sands materials. Toxicity of both solid-phase material (*e.g.*, fine tails, oil sands, reclamation materials) and liquid-phase (pond water, porewater, leachate, and groundwaters) have been studied.

2.3.1 Solid-Phase

Alberta Environmental Centre conducted acute and subacute toxicity tests on fish exposed to bitumen from the Athabasca River [23]. Exposed bitumen samples for the 1986 study were collected ~11 km downstream of Fort McMurray.

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The samples were separated, using an ultrasonic shaker, into three fractions: the whole bitumen sample, extractable bitumen, and residual bitumen. Acute toxicity of these fractions was assessed with the 96-hour rainbow trout LC_{50} test, and subacute tests included histopathology and blood analysis. The test solutions were prepared with the bitumen fractions added to treated municipal water to give concentrations ranging from 1 mg/L to 100 mg/L. The results obtained from the study concluded that the LC_{50} of all fractions were greater than 100 mg/L bitumen. Furthermore, there were no significant differences in the histopathology of the test fish compared to the controls. Blood analysis also supported the lack of significant changes between the test and control fish. The findings of this study indicate that exposure to naturally occurring bitumen along the Athabasca River is not acutely toxic to fish.

Golder Associates conducted a series of toxicity tests on solids and extracts obtained from solid-phase materials as part of a study to assess dry landscape reclamation options [24]. Overall, levels of toxicity of the solid-phase materials were low with respect to earthworm survival and seed germination. A decline in earthworm survival was recorded only for the oil sands sample, whereas effects on seed germination were reported for samples of oil sands, clay shale overburden from Syncrude's lease, and some fine tails samples. The dominance of phytotoxicity may have been related to the presence of phytotoxic substances, over which earthworms have more resistance, high salt levels, and/or differences in water potential (*i.e.*, many of the materials tested contained hydroscopic clays that have a high adsorptivity for water).

Of the tests used to examine leachate toxicity, nematode survival, seed germination, root growth, and SOS-chromotest showed no response while nematode maturation, algal growth, and Microtox showed a variable response. The different responses were likely a result of a number of interacting processes such as changes in nutrient, metal, and hydrocarbon content among the different samples.

There is a great deal of variation among species in terms of sensitivity to fine tails. Oligochaete worms and the leech *Nephelopsis obscura* were quite resistant and exhibited high mortality only when exposed to undiluted fine tails. At the other extreme, sensitive mayfly nymphs (*Hexagenia limbata*) and amphipods (*Hyalela azteca*) were killed by fine tails concentrations as low as 11%. There was no consistent difference in acute toxicity between the two sources of fine tails: Suncor fine tails were more toxic to some organisms, Syncrude fine tails to others. For most organisms, fine tails were acutely toxic and the difference between Suncor and Syncrude sources was small. Leeches were the only organism that ever survived in full strength tails, and only in those from Suncor [25].

2.3.2 Liquid-Phase

Most of the work on toxicity of oil sands materials has focused on the acute toxicity of wastewaters associated with the tailings/settling ponds. Results from acute toxicity tests on porewater and pond water are summarized in Table 2-5. These data suggests that *Hyalela* and rainbow trout are the most sensitive test species and *Daphnia magna* the least sensitive. Limited data from a recent experiment conducted by Gunter *et al.* indicate that *Ceriodaphnia* is also sensitive to process-affected waters [26]. In general, Syncrude's porewater and tailings pond water appear to be more toxic than water from Suncor, and water from the free water zone of the tailings pond at both facilities appears to be more toxic than porewater.

A number of experiments have been conducted to examine detoxification of tailings pond waters [27,28]. For example, where active input of tails material has ceased, a

TABLE 2-5. Summary of acute toxicity text results

Water source	Test			
	Microtox (IC_{50})	*Daphnia magna* (LC_{50})	*Hyalela azteca* (% survival)	Rainbow trout (LC_{50})
Tailings pond water				
Syncrude	22–43%[1,2,3,4,5]	76%[1]	–	12.5–13%[1,3]
Suncor	16–50[1,2,3,5,6]	98–100%[1,6]	–	3.2–28%[4,7,8]
Fine tails porewater				
Syncrude	20–40%[1,2]	–	–	–
Suncor	30–80%[1,2,6]	>100%[6]	10%[6]	–

[1] Reference [30]; [2] Reference [22]; [3] Reference [13]; [4] Reference [31]; [5] Reference [29]; [6] Reference [20]; [7] Reference [32]; [8] Reference [28]

marked increase in biological activity occurs and there is a reduction in toxicity. Survival of trout and Daphnia over a four-day test period in such pits increased from 0% at the start to 60–80% after 10 months [27]. Other tests have shown that acute toxicity to rainbow trout in top water in Suncor's Pond 1 is reduced from an LC_{50} of 7.5–10.2% initially to >100% after two years in test tanks in which phosphate has been added to the top water [28]. Dramatic decreases in chronic toxicity were also demonstrated. *Daphnia magma* lived an average of 4.8 days after pond water was treated for two years compared to less than one day for untreated Pond 1 water, and algal cells were able to grow compared with 100% mortality in untreated water. The ability of trout eggs to hatch and for the young fish (alevins) to survive was also substantially enhanced over this two-year period, compared with untreated top water.

EnviroTest and HydroQual carried out a number of laboratory experiments on rainbow trout to evaluate effects associated with long-term exposure (up to 29 days) to sublethal concentrations (1% solution) of topwater from Syncrude and Suncor's tailings ponds [29]. These tests included measuring the activity of detoxification enzymes, stress and reproductive hormones, chemical levels in tissues, and the general health and condition of the fish. The laboratory exposure tests indicated that levels of liver detoxification enzymes (mixed function oxygenases), blood plasma stress and reproductive hormones, PAH-metabolites, fish condition factors, lymphocytic ratios, and blood glucose increased following exposure to a 1% dilution of water from both Syncrude and Suncor's ponds.

In addition to the laboratory tests outlined above, EnviroTest and HydroQual carried out a number of experiments on rainbow trout held up to 77 days in Syncrude's experimental pits [29]. These experiments were designed to simulate conditions that fish would be exposed to in water-capped fine tails lakes (*i.e.,* levels of contaminants would be considerably lower that the 1% of pond water tested in the laboratory experiments). The fish were tested for the same suite of chemical and physiological variables outlined in the previous paragraph. The results of these experiments were inconclusive as no clear patterns in the measured variables were observed between the control pit and the test pits.

2.4 References

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3.0 WATER CAPPED FINE TAILS LAKES

3.1 Conceptual Basis

The wet landscape option for fine tails reclamation involves the storage of fluid fine tails in the mined-out pits. Conceptually, the wet landscape option disposes of mature fine tails as a fluid (>30 wt% fines), over which a layer of water is placed [1–3]. The objective of this reclamation option is to produce a stable, productive and self-sustaining landscape, which is integrated into the total reclaimed area, and meets the following criteria:

- geotechnically stable and non-erosive,
- productive capability at or above the pre-disturbance landscape, and
- free of long-term maintenance.

3.2 Environmental Considerations

3.2.1 Design Issues

3.2.1.1 *Lake Design*

The wet landscape option essentially leaves the fine tails as a fluid, which means that they are non-trafficable and require containment in geotechnically secure areas. The surface mining of the oil sands results in development of large mined-out pits, which may meet the criteria for areas to be used for long-term secure containment of fluid materials [4].

Containment within the mine pits would be below original grade and depths of more than 40 m of fine tails are expected, with the surface areas of the lake(s) being determining by the accepted mine plan [4]. During the period of fine tails transfer, further densification will occur (as a result of dewatering and consolidation), so that by the time a capping layer of water is applied, average solids content of the fine tails should be greater than 40%.

Syncrude has received endorsement from the AEUB for the development of a full-scale demonstration of water capping of fine tails [5]. This full-scale demonstration will involve transferring approximately 150×10^6 m^3 of MFT to a mined-out pit, and capping with a 5 m layer of water (33×10^6 m^3) to form a fresh water lake. The lake will be allowed to develop until it was acceptable, after which local runoff would be diverted into the lake and the outflow would be channeled into Beaver Creek.

3.2.1.2 *Resuspension*

The principle upon which the wet landscape approach is based is that the rheological properties of mature fine tails (low permeability, high density, relatively high yield strength and viscosity) are conducive to the success of the capping scheme [4]. As a result of these properties, the establishment of a well stratified aquatic system is possible, with two distinct layers (water and fine tails) formed with very low potential for active mixing of the two zones.

Lawrence *et al.* has investigated the potential for fine tails resuspension using laboratory flume tests [6]. These tests were conducted under various current and wave conditions to determine the energy required to disturb mature fine tails. Results showed that orbital velocities of waves of about 4 cm/s were required to break the interface. However, laminar mixing under currents require much higher velocities (>15 cm/s) to break the interface. In the proposed water capped lakes, the main source of these mixing energies would be generated by wave action, and hence would be a function of wind speed, fetch, wave height, and period. Based on historical wind records, there is a very low probability of the occurrence of storm or convective thermal mixing events capable of generating velocities required to suspend the fine tails under the planned water cap depths (Figure 3-1). For example, a capping depth of 5 m would be sufficient to prevent resuspension during a one in one hundred year storm for a lake with a fetch of 4 km.

3.2.1.3 *Capping Water Source*

The fine tails would be capped with water over a period of several years [4]. The source of the capping water would be determined by availability and limits set by ongoing research results and regulatory approvals. Potential sources include various combinations of one or all of the following: natural surface waters from local rivers or diffuse runoff, drainage waters from dry reclamation features (*e.g.*, sand storage areas), and/or process-affected waters.

3.2.1.4 *Evolution of the System*

Within one to two years of capping, it is anticipated that concentrations of organic compounds in the surface water will be reduced below levels that would be acutely toxic to fish and other biota (Figure 3-2) [7]. Over time, a detrital layer will build up over the fine tails layer, forming a natural lake sediment, which would be expected to act both as a barrier for potential mixing of fine tails into the capping water as well as a biologically active medium to enhance degradation and mineralization of compounds released by diffusion and consolidation of the fine tails [4]. It is, therefore, expected that the water-capped fine tails lakes will have the potential to develop into viable aquatic ecosystems shortly after capping.

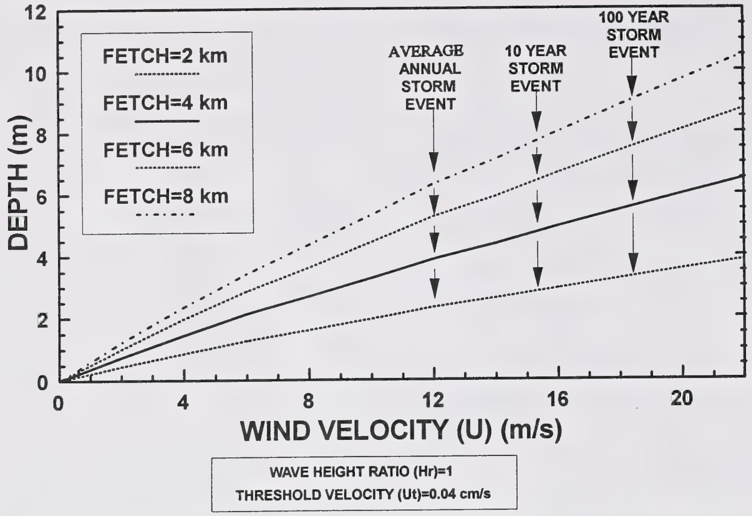


FIGURE 3-1. Relationship between the depth of capping water layer and wind speed for various fetches of water bodies [6].

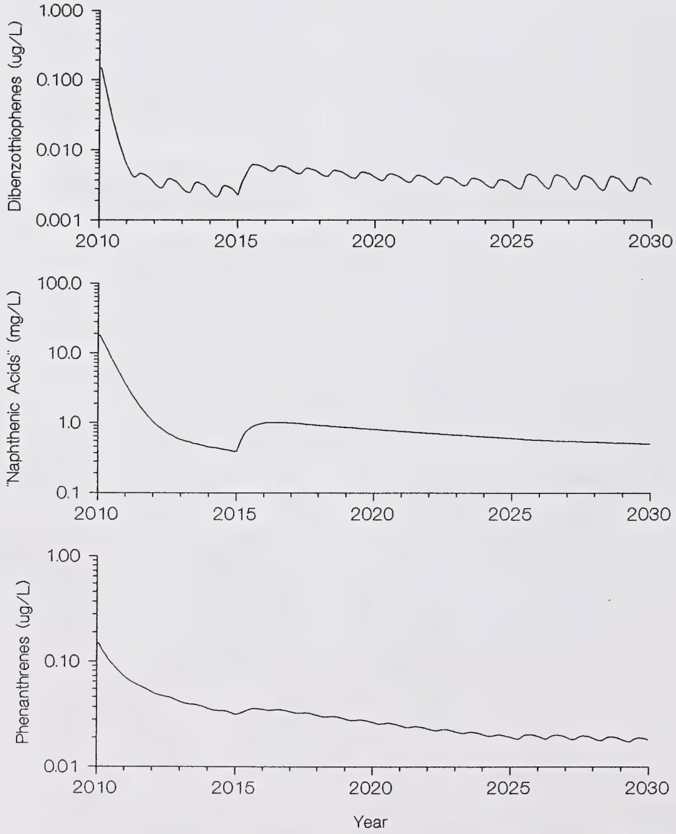


FIGURE 3-2. Predicted concentrations of organic contaminants in Syncrude's proposed Base Mine Lake [7].

There are a number of factors that will affect the development of the aquatic plant and animal communities [7]. For example, initially, light penetration into the lake will be limited by abiotic turbidity associated with the capping water. This turbidity should be reduced to acceptable levels once capping is complete and water current velocities are reduced (*e.g.*, during winter as a result of ice cover). Once the turbidity levels decline, it is expected that the lake productivity will be limited by the nutrient phosphorus. Phosphorus additions may be required initially to enhance bacterial growth (to ensure rapid biodegradation of organics) and to stimulate primary production. Over the longer-term, lake phosphorus concentrations will achieve a dynamic equilibrium with external and internal loads and lake processes, giving a projected average phosphorus concentration of ~7 μg/L and algal chlorophyll-*a* levels of 2-3 μg/L. These levels of phosphorus and chlorophyll-*a* are within the ranges reported for lakes of comparable depth in northern Alberta.

It is anticipated that with the exception of the first 20 or so years following capping, the lake will intermittently stratify during the summer months. Once the lake depth increases to more than ~10 m (as the fine tails dewatered), the lake will likely thermally stratify throughout the summer months until fall overturn [7]. With the possible exception of the first few years following capping, the lake should remain adequately oxygenated during the winter, thus, enabling the lake to support fish year-round.

3.2.2 Water Quality Issues

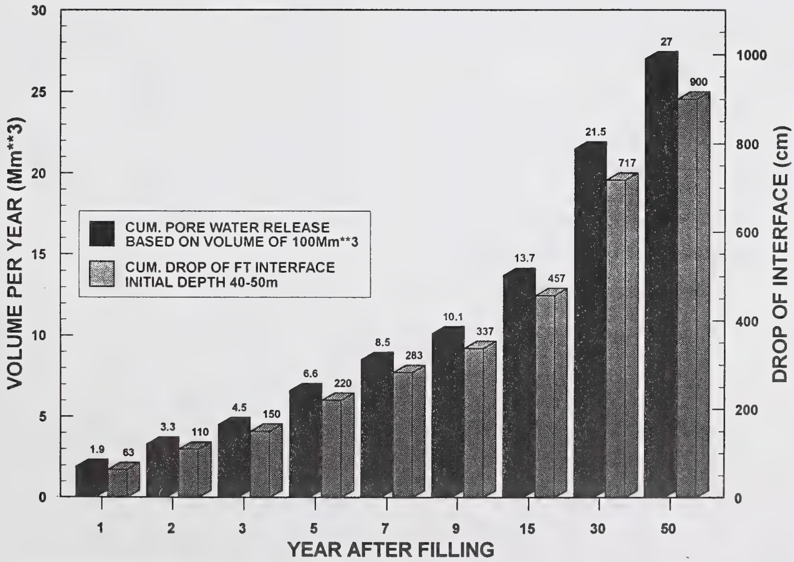
3.2.2.1 *Pore Water Release*

Even though the rate of densification of mature fine tails is slow, there will be a long-term release of porewater into the capping layer. The rate of release is projected to be highest in the first ten years following capping after which rates should decline over time (Figure 3-3). As a result of densification, the fine tails interface should drop an average of 10 cm/yr over the first 100 years, with the average loading factor of <1% per year of the original capping volume [8].

3.2.2.2 *Contaminant Fate*

As noted in Chapter 2.0, the expressed porewater will contains elevated concentrations of a number of chemicals and will be toxic to aquatic biota. Even so, results of both computer simulation studies and field experiments conducted in small-scale pits suggest that these water capped lakes will be capable of assimilating contaminants and that surface waters will support sensitive aquatic biota within a few years following capping [7,9].

Herman *et al.* examined the biodegradation of naphthenic acids by determining the biodegradation of carboxylated cycloalkanes and hexadecane in fine tails and porewater [10].



INITIAL FINE TAILS AT START=35.8% (f/f+w)
PORE WATER RELEASE PER 100Mm**3 WITH AREA 3km**2
ESTIMATED FOR LAKES OF ABOUT 40m DEPTH

FIGURE 3-3. Water release from MFT over time and change in depth to water-fine tails interface for Syncrude's proposed Base Mine Lake [7].

The carboxylated cycloalkanes used in that study, cyclopentane and cyclohexane, are structurally similar to naphthenic acids as they represent the carboxylated cycloalkane backbone of naphthenic acids (Figure 2-4). Further, they were used because a pure model for naphthenic acids does not exist (*i.e.*, naphthenic acids are available commercially only as a complex mixture of many different types of compounds). Mineralization of hexadecane was examined to provide an indication of the potential for biodegradation of the alkyl side chains of naphthenic acids (Figure 2-4).

Biodegradation of carboxylated cycloalkanes was rapid in fine tails porewater, but only after nitrogen and phosphorus had been added. For example, no degradation of cyclopentane or cyclohexane was recorded after 40 days in cultures without nutrient additions, whereas complete degradation of these compounds was evident within one week of nutrient additions. Methylated cyclohexane compounds were much more resistant to degradation. Differences in biodegradation potential were evident between porewater and fine tails microcosms. Degradation of carboxylated cycloalkanes was rapid in fine tails even without the addition of nutrients. In addition, methylated cyclohexane was degraded in the fine tails microcosms after addition of nutrients but was completely recalcitrant in porewater. These findings indicate the potential for oil sands tailings microorganisms to biodegrade the carboxylated cycloalkane ring component of naphthenic acids. In addition, hexadecane mineralization was rapid in fine tails, but only after nutrient addition. This indicates the potential for degradation of the alkyl side chain.

Fates of other contaminants associated with expressed porewaters generally fall into three groups: (1) soluble organics that are volatile and/or easily biodegradable, (2) compounds that are oxidized (*e.g.*, ammonia), and (3) compounds that adsorb onto particulate matter and/or precipitate as a result of changes in redox conditions (*e.g.*, arsenic).

3.2.3 Long Term Sustainability

As noted above in Section 3.2.1.4, it is anticipated that water capped fine tails lakes will develop into viable aquatic ecosystems. Within a few years of capping, the surface water should be of sufficient quality to support sensitive aquatic biota. Factors that directly affect primary productivity, such as nutrient and salt levels and light penetration are expected to be within the ranges reported for many lakes in Alberta. The lake is expected to remain well-oxygenated during the open-water season and, with the possible exception of the first few years following capping, the lake should not become anoxic over the winter [7].

Investigations into the long-term sustainability of the water capped lakes fall into two general categories: laboratory and field experiments and computer modelling studies.

3.2.3.1 *Laboratory and Field Experiments*

Both Syncrude and Suncor have conducted numerous laboratory experiments and have constructed experimental pits to mimic the proposed water capped lakes. In previous sections, we have discussed the results of laboratory studies that have been conducted to assess (1) toxicity of fine tails and pond and pore water (Section 2.3), (2) resuspension of fine tails (Section 3.2.1.2), and (3) contaminant fate (Section 3.2.2.2). Recent laboratory experiments have included investigations into nutrient limitation of algal biomass. The results of these studies suggest that phosphorus is the primary nutrient limiting algal growth in Syncrude's experimental pits and that additions of both phosphorus and nitrogen lead to higher algal biomass levels than phosphorus alone [11].

To assess the potential effects associated with the water capping option, Syncrude constructed experimental pits, ranging in size from 0.25 to 3.5 ha in area and up to 7 m deep [2]. Test pits were filled with 4–5 m of fine tails and capped with 2–3 m of natural surface water or pond recycle water. One pit, which represents a control, was simply filled with natural water. The rate of densification of the fine tails in Syncrude's pits was much slower than expected based on laboratory experiments, and the impact on surface water quality was minor (<5% change in major ion content). Acute and chronic toxicity tests conducted during January 1990 (the pits were filled in September 1989) showed no indication of toxicity to *Daphnia magna* (life cycle) or rainbow trout eggs. Further evidence of the lack of toxicity is that viable fish populations, as measured by number, fecundity, and age structure, are increasing in all the pits capped with natural water [2].

3.2.3.2 *Computer Model*

A modelling framework (the reclamation landscape model) has been developed to assist in planning for oil sands mine reclamation (Section 6). A major component of this model is a module that simulates hydrodynamic, water quality, and biological conditions in a water-capped, fine tails lake [9,12]. This Wet Landscape Module can be used to predict water and sediment concentration of eutrophication-related variables (dissolved oxygen, nitrogen and phosphorus fraction, chlorophyll-*a*), conservative variables such as some major ions and total dissolved solids, and organic compounds such as naphthenic acids and PAHs. In addition, concentrations of organic contaminants in aquatic biota (including fish) can be predicted.

The Wet Landscape Module has been tested against water quality data collected from both Syncrude and Suncor's experimental ponds (Figure 3-4). In addition, it was used during Syncrude's ERCB hearings to assist in evaluating water quality conditions in the proposed Base Mine Lake, *e.g.*, for predicting levels of organic contaminants in the lake (Figure 3-2) [7].

3.3 References

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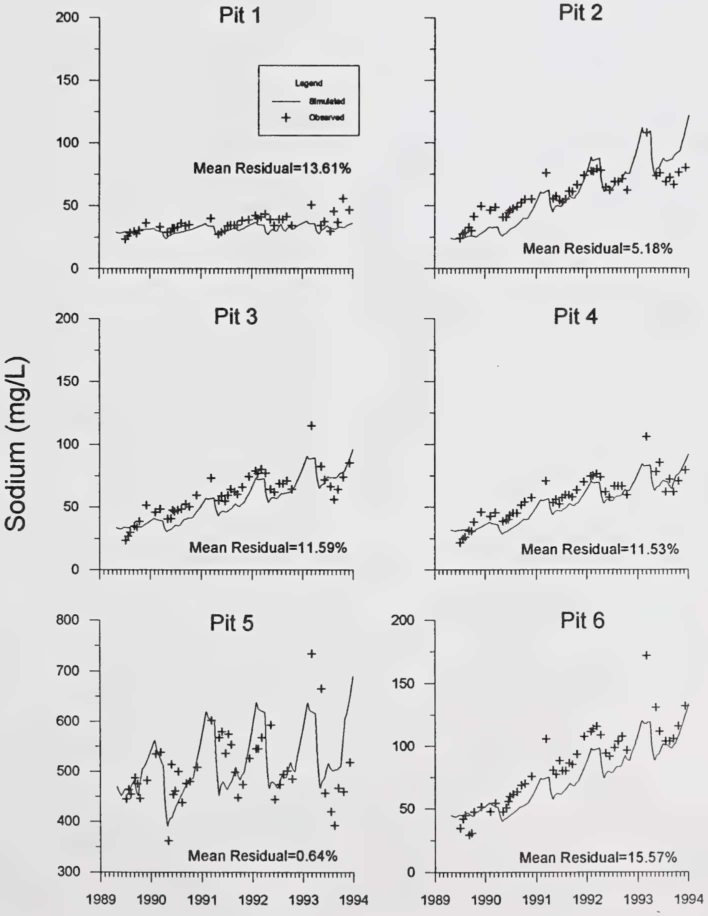

FIGURE 3-4. Predicted *versus* observed sodium concentration in Syncrude's experimental pits [9].

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4.0 DRY LANDSCAPE OVERVIEW

4.1 Conceptual Basis

The dry landscape options aim to produce a solid deposit which would ultimately be trafficable and capable of being capped with soil for terrestrial vegetation and reclamation [1]. Some of the various dry landscape options include:

(a) dewatering of the fine tails to a more fully consolidated clay through processes such as evaporation and freeze-thaw;

(b) incorporation of fine tails with overburden clays (adsorption of fine tails pore water with clay mixes) and with increased fine tails solids within coarse tailings sand; and

(c) non-segregating or consolidated tails; that is, lime, acid/lime, or calcium sulphate addition to extraction tails.

Although a large data base was surveyed, literature relevant to the environmental implications of dryland reclamation of fine tails in the mineral industry is very limited [2]. Most of the case studies in the published literature relate to the management of oily sludge from refinery operations. The impacted soils are generally of higher oil content but are of less volume overall than fine tails from the oils sands process.

Detailed descriptions on engineering technologies designed to create dry landscapes are available in Volume III of this final report.

4.2 Environmental Considerations

Dry landscape reclamation alternatives primarily involve production of an artificial subsoil through incorporation of tails within a soil matrix or consolidation of the fine tails to a soil consistency [2]. Since the end result is to be a functioning terrestrial ecosystem, the latent toxicity or inhibitory effect of the fine tails after incorporation into a dry landscape must be evaluated. Off-site impact assessment and mitigation of dry reclamation strategies must consider migration of contaminants to surface waters *via* direct runoff or groundwater pathways; aerial migration of contaminants *via* air or dust, and the potential for food chain biomagnification. Direct or indirect impacts of contaminant migration on human populations must also be considered (Figure 4-1).

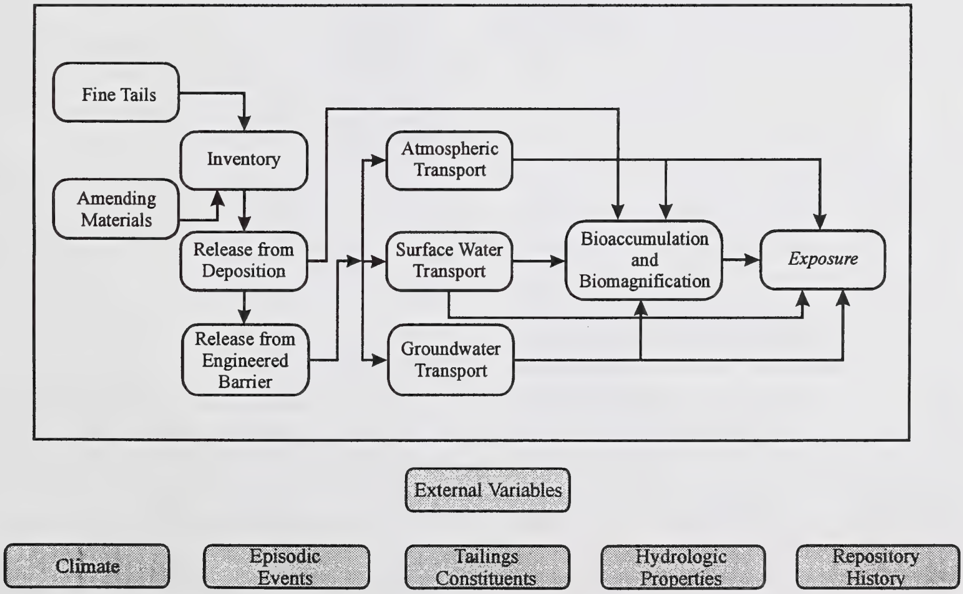

FIGURE 4-1. Dry landscape reclamation: Pathways Model, from Environmental Management Associates [2].

Traditionally, site assessment for the remediation of plant sites (not mines) relied on specific criteria set out by Alberta Environmental Protection in the license to operate. More recently, however, chemical-specific guidelines such as the hazardous waste criteria, Alberta Tier 1 criteria, and surface reclamation guidelines for oil sands facilities are being adopted [3]. In an argument to include toxicological testing for the evaluation of dry land reclamation techniques, the authors conclude that

> "strict adherence to the use of chemical specific criteria for reclamation assessment can be limiting in that the criteria are general in nature and cannot account for site specific phenomena which could affect the bio-availability of a contaminant or potential for leaching into the surrounding geology and aquifers. Secondly, the chemistry approach can be inadequate for assessing potential synergistic effects of a complex mixture."

As a response to these issues of assessment protocols for reclamation, protocols have been developed for the chemical and toxicological testing of fine tails using "soil" column testing in the laboratory [2]. In the field, Suncor established soil plots to provide the capability for long-term and multi-faceted environmental studies (*e.g.*, leachate quality, vegetation growth, soil micro-organisms) to assess the environmental impact of fine tails amendments to soil [4,5].

4.2.1 Stability

4.2.1.1 *Soil Amendment*

Although fine tails are toxic (using the Microtox bacterial assay) when first applied to soils, the organic constituents will be biodegraded and can be expected to ultimately detoxify (in aerated systems). Biodegradation processes can be assisted by careful management including an annual fertilization program. Leaching of contaminants to groundwater should not be a serious problem if the areas used for land treatment are small and/or are staggered over time among adjacent "cells." The effects on groundwater quality from the application of fine tails to large areas are not known (since groundwater tends to lack air and hence biodegradation processes would likely be inhibited). The results from this study support previous recommendations that the "low" range (5 to 6 cm depth) of fine tails application shows promise, but that higher loading rates could be difficult to manage [5]. Also, the total area of land needed to dispose of the total available quantities of fine tails in this manner is vast and functionally impractical. It was concluded that the environmental risk for land treatment of fine tails on groundwater and surface water quality is manageable. Pilot-scale field assessments would help to verify this and would be necessary to refine the management practices required for full scale land treatment.

4.2.1.2 *Freeze-thaw*

A considerable amount of water is released when thin layers (5 to 15 cm) of mature fine tails are subjected to freeze-thaw cycles. For 15 cm thick layers, thaw strain in excess of 50% was apparent for all the temperature boundary conditions investigated. The decant water contained less than 0.5% solids. Settled solid contents were 45 to 55%. A volume change of 60% was realized for 30 cm thick layers [6]. These authors concluded that freeze-thaw tails would effectively support a sand blanket provided sand placement is achieved *via* sedimentation through water.

Experiments conducted in laboratories and in small field tests demonstrated that a freeze-thaw cycle can increase solids concentrations of fine tails from 30 to 50 wt% [7]. However, in the field, the freeze-thaw option would required large areas of land. To treat 21 Mm^3/year of fine tails, at an assumed optimum depth of 3 m, a total of 7 km^2 of surface land would be required [8].

4.2.1.3 *Sand Layering*

Although a thin sand layer could be established on fine tails having a solids content in the 24 to 28% range, the layer would consistently prove to be quite unstable and failures, when initiated, would tend to progress across the entire deposit. With a solids content greater than 30% and with aging of the deposit prior to layering, significant differential loading across a layer could be supported and a relatively thin layer would exhibit significant resistance to progressive failure induced by layer penetration [9].

4.2.2 Expressed Water

Inherent within all above grade dry landscape options is the requirement to control waters that will be draining, leaching, or running off reclamation areas because these waters may contain unacceptable constituents of the fine tails materials [1]. Dry land reclamation schemes may result in the production of substantial volumes of leachate as fine tails interstitial pore water drains from the soil. Preliminary research [10] has shown that even after mixing fine tails with soils, leached water can exhibit some toxicity to aquatic organisms and hence may require treatment. The environmental fate of contaminants from expressed water in dry land reclamation trials has not been extensively investigated. Since the water quality of this water would likely be comparable to Suncor's dyke drainage water, the best assessment of any environmental impacts of released water would be through a review of the wetlands research (*cf.* Volume II — Chapter 5.0).

4.2.2.1 *Fine Tails Burial*

One concept for the disposal of fine tails below grade is to incorporate or bury them with various combinations of overburden materials [11]. In the development of a dry landscape, water will be released from the tails deposit during consolidation. Some of the pore water in the consolidated tails may leach out in direct runoff or groundwater. Since the pore water contains various hydrocarbons and other organic compounds, and if migration from large "burial pits" occurs, it may have significant effects on the surrounding terrestrial ecosystem [12]. Conclusions from a study of leaching column tests on a variety of materials included:

(a) Most primary (*i.e.*, initial) leachates were slightly to moderately toxic to bacteria but strongly toxic to algae.

(b) Genotoxicity assays produced mixed results, with some evidence of an adverse impact, most notably for Suncor tails which were strongly inhibitory to nematode maturation.

(c) Metals of concern included aluminum, cadmium, iron, manganese, and zinc; however, their toxicity was less than expected based on a review of the literature. Copper and some other heavy metals were of concern in some samples. It was noted that metal concentrations in leachate may not decline over time; indeed, some increases were observed in subsequent pore water replacements. Aluminum and iron appeared to be derived from sand, rather than from fine tails; however, acid/lime treatment of fine tails (*i.e.*, consolidated or non-segregating tails) released both of these metals, but freeze-thaw did not.

(d) There was no correlation between contaminant concentrations and the degree of toxicological response and also none between conductivity and algal inhibition, indicating the need for a suite of tests.

(e) Finally, there was a concern that high levels of phosphorus might result in nutrient enrichment of runoff water and subsequently in water bodies in the receiving environment.

These results were comparable with leachate quality following a soil amendment scenario (*cf.* Section 4.2.4) in terms of identifying phosphorus as a compound of concern. However, the indication that some metals might increase in concentration over time was not identified by Hamilton and Shum [10].

4.2.2.2 *Dewatering*

Freeze-thaw interstitial water from the Suncor site had a decreased level of toxicity compared with pond top water, using the microtox bacterial assay and trout LC_{50} tests [13]. However, its toxicity to the aquatic invertebrate *Daphnia magna* was comparable with that for tailings pond top water.

In terms of hydrocarbon content, freeze-thaw water had a TEH (total extractable hydrocarbon) concentration of just 15.5 ppm compared with 98,400 ppm for untreated fine tails [13]. In a comparison of GC/MSD (gas chromatography/mass selective detection) profiles, a characteristic "naphthenic acid peak" was determined for the freeze-thaw water, indicating that contaminants characteristic of those naphthenic acid-like compounds in fine tails would be released to the environment, albeit in vastly reduced concentrations. Many of the higher molecular weight hydrocarbons were not released into water produced by freeze-thawing of the fine tails.

4.2.2.3 *Evaporation*

A field study used bermed terraces and disked furrows to assess the success of dewatering fine tails using natural evaporation [14]. Dewatering of fine tails to 70% solids resulted in a 30% volume reduction. The authors noted advantages to this technique with respect to a reduction of wind erosion of the surface of the tailings sand. There was no analysis of the fate of contaminants in released water or the remaining solids.

4.2.3 Contaminant Fate

With the exception of soil amendment scenarios, there has been little research to investigate the fate of contaminants (*e.g.*, uptake in plant or insect tissue; mineralization, volatilization).

4.2.4 The Suitability of Fine Tails as a Growing Medium for Plants

4.2.4.1 *Soil Quality*

Incorporation of fine tails into soil mixtures during the reclamation of disturbed areas has been proposed as one option for its ultimate disposal. In 1979, Suncor initiated a study to assess the potential value of fine tails as a soil amendment on reclamation sites [4]. The tailings sand within dykes and other areas is generally a poor medium for plant growth due to poor structure, low nutrients, and poor pH buffering capacity [4]. Peat (muskeg) may be spread over the surface prior to seeding to compensate for these limitations. Although the peat adds much needed nutrients and water holding capacity to the soil, peat additions do not significantly improve soil structure due to an absence of clay. As a result, fine tails was proposed as a soil amendment, primarily because of its high clay content. A five-year (four growing season) study was undertaken to compare standard reclamation procedures (peat alone) with treatments where overburden or fine tails was added with the peat [15,16].

The emphasis of these studies was the potential of the fine tails as a soil amendment, rather than an assessment of dry reclamation *per se*. They concluded that fine tails was not a viable source of mineral fines since revegetation was hampered by high sodicity and high bitumen levels [16]. Serious material handling problems also existed due to its thick consistency, slow dewatering rates, and characteristics which made it difficult to mix with other components to form a soil. Plant tissues in study plots with fine tails demonstrated more nutrient deficiency symptoms, especially for nitrogen, compared to other treatments. However, these detrimental effects appeared to diminish with time possibly suggesting that some level of fine tails amendment is possible. The relatively short duration of these experiments made it difficult to make definite conclusions with respect to long term revegetation success and soil stability.

Eleven years after the initial study, an EVS study assessed the long-term impacts of fine tails amendments to soils and vegetation [5]. Three treatments were evaluated: no fine tails (control), low fine tails (5.5 cm depth), and high fine tails (12 cm depth). All soils had been amended with 15 cm of muskeg. The plots were sampled for various soil (hydrocarbons, metals, nutrients, chemical/physical properties, microbial activity) and vegetation (above-ground biomass, root mass, cover, nutrient and metal contents) parameters. As well, soil toxicity testing was carried out using a nematode (*Panagrellus redivivus*) bioassay.

The vegetation above-ground biomass and percent vegetation cover were significantly lower on those plots which received the fine tails amendments. This was partly explained by differences in available nutrients in the soil, particularly nitrate, which may not be readily available to plants in the fine tails plots due to their preferential utilization by hydrocarbon-degrading microorganisms (levels of hydrocarbons were significantly higher in the soil amended with fine tails). Also, the effects of differential fertilizer applications on the plots is still apparent, despite there being eight years since fertilizers were last applied. Soil toxicity test results were somewhat mixed, but suggested some residual toxic effects of the fine tails applications.

The addition of fine tails to reclaimed soils continued to have a negative impact on plant productivity, 11 years after application [5]. However, the magnitude of this negative effect had diminished considerably since 1980–83. The data indicate that any differences between soil with and without fine tails as a supplement may be further diminished and/or eliminated over longer periods as a result of progressive bioremediation (*i.e.*, microbial decomposition of oily residues), leaching, and a reduction in the residual effects of fertilization.

The ability of reclaimed soils to successfully incorporate fine tails as a supplement requires further research to assess long term impacts (*i.e.*, greater than 11 years). However, on the basis of one study [5], it is considered likely that an optimum loading rate would be a depth of about 5.5 cm.

A comprehensive rating system has been developed [2] to assess reclamation suitability for a variety of fine tails, treated by different methods (*e.g.*, freeze-thaw, acid/lime) or in combination with various other substrates (*e.g.*, sand, topsoil, clay) and using a wide array of toxicological and chemical tests (Table 4-1). The rating system utilized a wide suite of chemical (*e.g.*, metals, hydrocarbons, conductivity) and toxicological (*e.g.*, earthworm survival, seedling emergence, root elongation, algal growth, genotoxicity) tests to assess overall environmental acceptability.

This evaluation was considered tentative since ratings by nature tend to minimize specific areas of concern, since there were limited comparisons with reference materials, and since other factors relevant to "reclaimability" were excluded (*e.g.*, soil structure). They concluded that many types of "treated" fine tails would provide a "good" rating as reclamation material. Interestingly, the amended tails (*i.e.*, fine tails plus tailings sand) tended to have higher toxicity ratings and lower chemistry ratings than many unamended tails. This apparent contradiction was unexplained.

4.2.4.2 *Leachate Quality*

In a laboratory study, it was found that for most compounds in the leachate (using soil columns with mixture of sand, muskeg, and fine tails), there was a marked reduction in chemical concentrations found in the second six-day period compared with the first [10]. These compounds include oil and grease, conductivity, iron, zinc, aluminum, nitrate, ammonium, and the exchangeable cations (K, Ca, Mg, Na). The only element which showed a consistent increase in the second cycle was phosphorus. The oil and grease content of one sample was higher in the second cycle, but this probably reflected the very low hydraulic conductivity.

TABLE 4-1. Summary of ratings for reclamation material for different combinations of tails, from Environmental Managements Associates [2]

Sample no.	Material	Source	Overall solid toxicity	Overall solid chemistry	Overall leachate toxicity	Overall leachate chemistry — water extract	Overall leachate chemistry — weak acid extract	Overall rating as reclamation material
	Reference materials:							
1	Control soil	Hydroqual	N	N	N	N	n/a	Excellent
15	Topsoil	Suncor	N	n/a	N	M	M	Good
2	Overburden	Syncrude	M	M	M	M	H	Fair
3	Oil sand	Syncrude	H	L	N	L	H	Poor
	Sand:							
7	Tailings sand	Suncor	N	M	M	N	M	Fair
11	Tailings sand	Syncrude	N	H	N	N	n/a	Good
	Fine tailings:							
4	Dry Pond 1 test pit	Suncor	N	L	M	L	H	Good
5	Dry Pond 1 beach	Suncor	M	N	N	L	n/a	Good
10	Air dry	Syncrude	L	M	H	H	H	Poor
12	Freeze/thaw	OSLO	L	N	N	N	H	Good
13	Consolidated	OSLO	L	L	N	N	n/a	Good
	Amended tailings:							
6	Freeze/thaw	Suncor	M	N	N	L	H	Good
8	Sand/fine tails	Syncrude	M	L	N	H	H	Fair
9	Clay/fine tails	Syncrude	H	H	N	H	H	Poor
14	Acid/lime treatment	Suncor	H	n/a	n/a	H	H	Poor

Rating scale (degree of concern): N = Negligible, L = Low, M = Moderate, H = High.

From the perspective of water quality, only three metals (Fe, Zn, and Al) exceeded the Canadian Water Quality concentration guidelines for aquatic habitat. All showed declining concentrations in the second cycle thus suggesting an ultimate reduction below guidelines. Field monitoring would be required for these and any other compound to ascertain that concentrations continue to decrease. The ongoing cycles of physical and chemical weathering in the soils could cause certain elements and compounds to have renewed availability. If followed by a flush of gravitational water, such as during spring snowmelt, there could be a temporary increase in concentrations.

Beyond the question of concentrations, there is the issue of contaminant loadings. The actual volumes of leachate which can be expected to move through the soil profile is small, due to a combination of moderate rainfall, summer evapotransformation, and the very low saturated hydraulic conductivity of the fine tails amended soils. Thus contaminant loadings (load = volume × concentration) may also be relatively low. Any leachates moving to groundwater would be diluted by regional groundwater flow before moving to the Athabasca River or other surface water bodies. This anticipated situation could change if large areas are utilized for land treatment, or if multiple fine tails loadings are applied to a single site. This could be managed by applying the fine tails in block areas or cells, and spacing the timing of applications in adjoining cells.

4.3 Long Term Sustainability

4.3.1 Modelling

A conceptual and analytical framework for quantifying risks to terrestrial wildlife that might be exposed to soil-phase materials potentially associated with oil sands reclamation has been developed [17]. The initial phase of the assessment involved screening the chemicals measured in various soil-tails samples against published criteria to produce a short list of 10 chemicals (eight organics and two inorganic) that pose a potential hazard to terrestrial biota. A probabilistic model was developed to (1) simulate exposure doses to three representative terrestrial wildlife receptors (deer mouse, white-tailed deer, American kestrel), (2) compute the probability of exceeding a chemical exposure limit for each receptor, and (3) summarize the relative contribution of the different exposure pathways (*i.e.*, food and water ingestion, incidental soil ingestion, inhalation) to the total exposure dose. Due to the paucity of data, a number of conservative assumptions were applied in this study that precluded firm conclusions with respect to potential risks associated with each of the soil-tailings samples. Nonetheless, the preliminary findings indicated that risks to white-tailed deer (and potentially to other herbivores) would be low, whereas risks to omnivores and carnivores may be higher as a result of ingestion of potentially tainted prey and incidental ingestion of potentially contaminated soil. Other exposure routes, such as ingestion of water and inhalation of air vapors are not expected to pose a risk for the chemicals evaluated in this study.

4.3.2 Land Use Options

The conceptual types of materials that could be utilized in both wet and dry land reclamation are shown in Figure 4-2. For dry land reclamation, options include: entrainment of the fine tails in overburden; accelerated dewatering by chemical, mechanical, and/or electrical means; and aggressive drainage *via* natural drying, freeze-thaw, and/or land farming [11]. Hence dry landscape land use options would include

(a) incorporation of dewatered fine tails into overburden to form dykes, or

(b) various forms of burial by which the underlying land would be formed with this material, perhaps including small lakes or wetlands, at the surface, to contain any released water.

4.4 References

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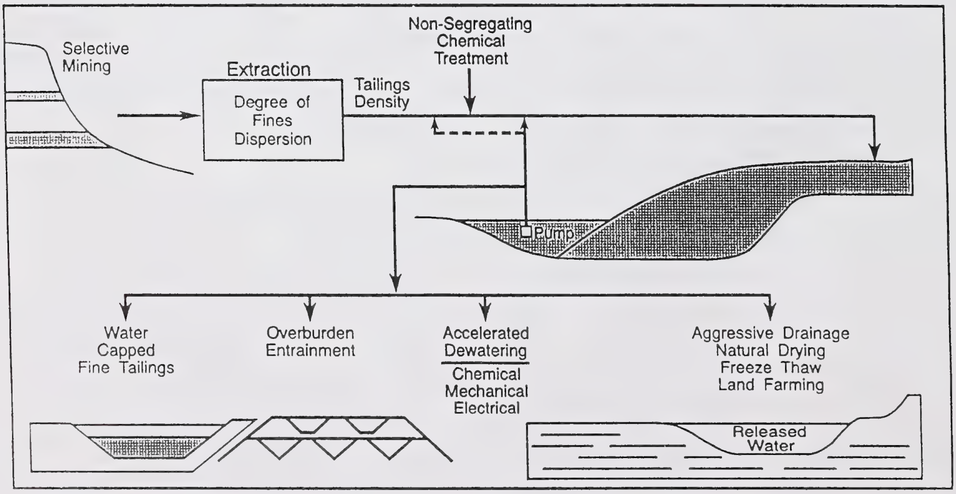

FIGURE 4-2. Fine tails volume reduction opportunities, from Proceedings of Oil Sands — Our Petroleum Future Conference [11].

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5.0 WETLANDS

The use of both natural and constructed wetlands as a means to treat contaminated water is emerging as an important technology [1]. The technology had its origins in observations of improvements in the quality of waters flowing through natural wetlands. Constructed wetlands are presently being used to treat a range of polluted waters including domestic wastewater, agricultural and urban storm runoff, acid rock drainage, mine waters, industrial discharges, and landfill leachates. Constructed wetlands also have important ecological and aesthetic functions, especially in areas where wetland coverage has been replaced by development.

The use of constructed wetlands was considered preferable over natural wetlands since [1]:

(a) there is evidence that designed and constructed wetlands perform better than natural wetlands in removing pollutants, as a result of superior control over flow, retention time, and vegetation mix [and other important parameters];

(b) although natural wetlands have been used to treat wastewater (*e.g.*, [2]), they are multi-function ecosystems and the discharge of effluent could have a negative impact on other functions (*e.g.*, wildlife habitat, recreation, aesthetics, flow regulation);

(c) construction creates additional wetlands in areas where the amount of wetlands space may have been reduced due to development pressure [3] or drought [4]; and

(d) some natural wetlands are considered waterways and the discharge of effluent into them may require a permit.

A constructed wetlands is defined as a designed and human-made complex of saturated substrates, emergent and submergent vegetation, animal life, and water that simulates a natural wetlands for the treatment of wastewater [5]. A schematic of its treatment capabilities is given in Figure 5-1. The term constructed wetlands is preferred over "artificial" wetlands since they perform as real, functional wetlands. Constructed wetlands range from low-technology systems where existing ditches or ponds have been only slightly modified, to systems that have been designed in detail and require a significant amount of construction. Most constructed wetlands simulate natural marshes in terms of the flow of water and the species of vegetation present. The basic design of constructed wetlands for oil sands reclamation work has involved surface flow wetlands (*i.e.*, wastewater flowing over sediment) since the large volumes of water involved would likely make subsurface flow wetlands (*i.e.*, wastewater flowing through a porous media such as sand or gravel which has been placed on the bed of the wetland) prohibitively expensive.

In the U.S., the EPA have encouraged communities to create constructed wetlands for treating stormwater [6]. In Canada, the use of wetlands for wastewater treatment systems is compatible with the recent adoption by the federal government of The Federal Policy on Wetlands Conservation in several aspects [7]:

(a) a recognition of wetlands functions in resource planning, management, and economic decision-making, and

(b) the enhancement and rehabilitation of wetlands in areas of continuing loss or degradation.

As a result of these policies, wetlands (*i.e.*, marshes) are increasingly being used to treat a variety of wastewaters, including municipal, agricultural, and industrial sources [8].

5.1 Conceptual Basis

The reclamation of fine tails material from oil sands mining and extraction plants will be accomplished through a combination of dry and wet landscape options [9]. Some water discharge from both of these fine tails reclamation approaches will occur: dry areas may have seepage of entrained or pore waters from below grade, groundwater discharge as part of the hydrological cycle, or runoff/drainage from the reclaimed areas; wet areas will have above grade water discharge from any lake or water capped system because of inevitable (and desirable) inflows of surface water runoff or groundwater, and below grade discharges to groundwater. Any water released from either wet or dry reclamation areas will likely require some amount of treatment since it may have a harmful impact on either aquatic organisms or terrestrial plants and animals by virtue of the presence of organic (*e.g.*, naphthenic acids, hydrocarbons) and inorganic contaminants (*e.g.*, ammonia) [7]. Therefore, both wet and dry reclamation approaches have included wetlands as an integral component of the reclamation process [10].

The results of preliminary work at the Suncor site, using dyke drainage water as representative of oil sands wastewaters, suggest that constructed wetlands may provide an innovative, cost-effective, environmentally acceptable alternative for the treatment of oil sands wastewater [11]. As well as acting as a passive buffer system between reclamation units and the receiving environment, a wetlands system would act as an active bioreactor treatment system for the retention or degradation of potentially deleterious contaminants [12]. However, site-specific research was necessary because, although constructed wetlands are well established as a means of treatment for domestic wastewater, this biotechnology has not been well developed for the treatment of industrial effluents and/or runoff water from sources such as reclaimed tails ponds or fine tails disposal areas.

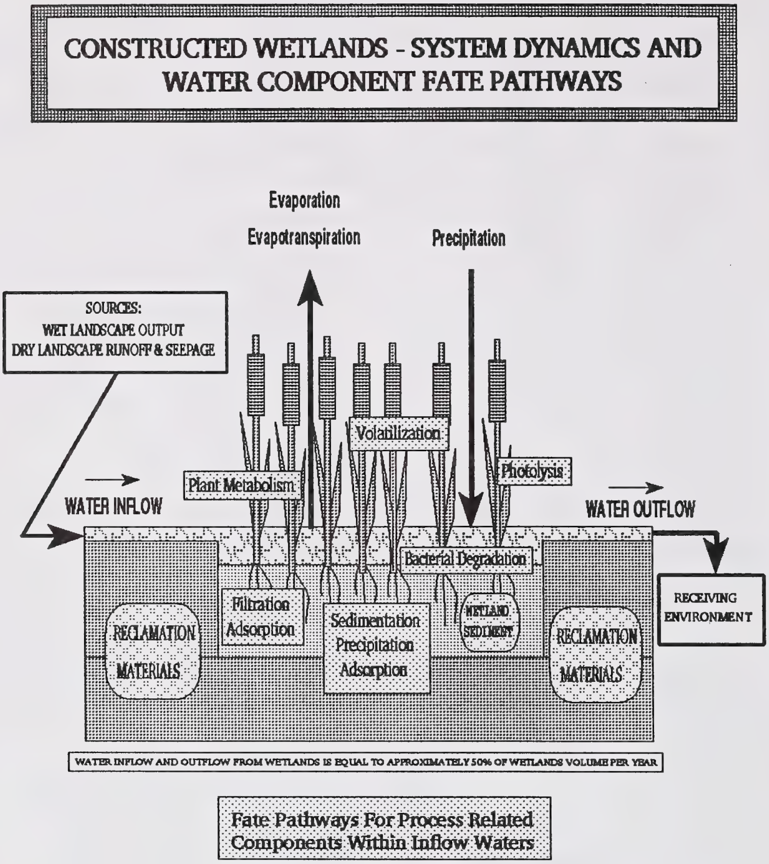

FIGURE 5-1. Fate pathways for contaminants in a wetlands treatment system.

The overall objective of this wetlands research has been to demonstrate the capabilities of constructed wetlands as long-term, "self-sustaining" (*i.e.*, naturally self-managed) systems for the treatment of large quantities of wastewater anticipated to be released after mine abandonment, or dyke drainage during the operational phase of the mine. The focus on a "self-sustaining" treatment systems (*i.e.*, with no ongoing manipulation) means that no structures or devices requiring routine ongoing maintenance should be left on the lease after mine abandonment. Notwithstanding this goal for a self-sustaining treatment system, events during that last three years have included the development of tails treatment processes which would release volumes of water which may require immediate treatment (*e.g.*, Consolidated Tails or CT water). The need for more rapid treatment technologies, and the apparent long hydraulic retention time (HRT) required for self-sustaining wetlands, have indicated the need for a more structured approach to treatment. Therefore, the research focus has been shifting from exploring the capabilities of self-sustaining wetlands to an assessment of "managed" wetlands treatment system; that is, various modifications to a self-sustaining wetlands such as phosphorus and/or aeration supplementation.

Overview

Initial investigations of Suncor's tailings ponds have indicated that, although the water was acutely toxic to fish (*i.e.*, mortality within 96 h), the surface layer of water did contain populations of bacteria, phytoplankton (mostly ultraplankton), and even a few species of zooplankton [13,14]. Furthermore, research has shown that contaminants within Suncor or Syncrude tailings ponds/settling basins are biodegradable and that the numbers and types of aquatic organisms that can colonize this water greatly increases during treatment in large ponds [15,16]. Also, phosphorus and aeration would act to stimulate the treatment process [17,18]. As a result of all these factors, biological treatment (such as wetlands) were recommended as a method of reclaiming or disposing of oil sands wastewater in 1985 [19].

Specifically, wetlands may be required to act as:

(a) passive treatment systems to improve the quality of water discharged over the short-term (*e.g.*, release water from freeze/thaw processes) or long-term (*e.g.*, releases from water capped lakes either directly as a discharge or indirectly as seepage through dykes);

(b) active treatment systems to permit the immediate discharge of oil sands wastewater into the aquatic receiving environment during mining operations and thereby reduce the volume of water required to be stored on-site and released gradually;

(c) aquatic buffer zones to disperse any impacts (*e.g.*, physical, chemical, or biological) from the release of treated wastewater to the environment; and/or, as a contingency holding/treatment system in the event of a temporary failure of a primary treatment system (*e.g.*, aeration ponds).

Reclamation of Oil Sands Wastewater

Various conceptual considerations regarding the use wetlands in the oil sands area have been considered [19] and are summarized as follows.

Self-Sustaining versus *Managed*

Self-sustaining wetlands are defined as passive systems (*e.g.*, natural systems with no manipulation of any kind) that might be used after mine abandonment.

Managed wetlands, as an alternative, imply an active treatment approach such as the addition of nutrients or aeration. These amendments would enhance the microbial degradation of contaminants within a wetlands, perhaps useful during the operational phase of an oil sands mine. A managed wetlands would offer several advantages with respect to the treatment of oil sands wastewater [19]:

(a) Since the anaerobic degradation of petroleum hydrocarbons is negligible compared with aerobic processes [20], aeration would likely stimulate the biodegradation of hydrocarbons. Aliphatics or a simple straight-chained hydrocarbons are degraded through general biochemical pathways; aromatics or complex hydrocarbons are additionally modified until their ring structures are opened, whereupon they are similarly degraded through these biochemical pathways [21]. The overall biochemical process of mineralization may be viewed as:

$$\text{organic material} + O_2 \rightarrow CO_2 + H_2O + \text{new organisms} + \text{heat}$$

(b) In bench-scale studies, phosphate was required to detoxify Suncor pond top water within 7 to 10 weeks at 20°C [17]. In later studies, the addition of phosphate in field-scale trials has shown an enhanced ability to detoxify Suncor dyke drainage water with a concurrent enhanced rate of nitrification; that is, the microbial oxidation of ammonia-ammonium [7,19].

Treatment Performance versus *Ecological Characteristics*

Treatment performance defines the capability of a wetlands to remove contaminants over a certain hydraulic retention period or HRT. In managed wetlands treatment systems, treatment performance is the primary consideration for the design of a wetlands. Indeed, a treatment wetlands would be incapable of functioning if its biological characteristics were identical to nearby natural wetlands [19]. For example, different microbial biofilm populations are necessary to biodegrade oil sands contaminants compared with populations in natural wetlands, and these differences at the level of primary production will inevitably be reflected in differences in both the types and numbers of other organisms in the aquatic food chain (*e.g.*, phyto- and zooplankton, benthic invertebrates).

The ecological characteristics of treatment wetlands become increasingly important as the size of the total wetlands area increases or with the degree of permanency of the wetlands as part of the overall landscape. For example, if a treatment wetlands occupies many hectares of land, then it will inevitably become an important ecological component of the landscape in terms of both terrestrial wildlife and waterfowl. Similarly, if this wetlands were to exist for long periods, continuing as dominant features of the landscape even after mine abandonment, then its ecological characteristics would need to be considered in terms of any long-term environmental impacts (negative or positive).

Pond versus *Marsh*

Wetlands may consist of both open water (pond) areas with water depths of greater than 1 m and marsh-like areas with more shallow water and higher densities of aquatic plants (macrophytes). Both areas have advantages in terms of treatment performance or ecological characteristics. For example, open pond areas have been recommended to enhance the microbial oxidation of ammonia-ammonium (*i.e.*, nitrification) [19].

Alternatively, areas with abundant surfaces in the form of plant stalks would likely enhance the potential for a bacterial biofilms which could then more rapidly degrade organic contaminants such as naphthenic acids [22]. Biofilms developed on plants and detritus would be much more resistant to sudden changes in the types or concentration of contaminants compared with planktonic (free-floating) bacteria. As a result, wetlands will sustain shock-loading much better than most engineered systems [1].

5.1.1 Natural and Constructed Wetlands

Suncor has undertaken primary research since 1991 on the use of wetlands as a treatment system for oil sands wastewater.

Natural Wetlands

The first experimental wetlands system at the Suncor site, referred to as the Natural Wetlands, is a small (12,770 m^2) wetlands area which developed within a reclamation area on a level tails sand section south of an existing Suncor tails pond. This area had developed under the influence of waters from three primary sources: tails dyke seepage waters, groundwater, and area precipitation and runoff [10]. Since 1992, modifications to enhance the Natural Wetlands as a research area have included: installation of a weir to increase water levels and HRT; construction of berms to promote mixing and minimize channelization; and the installation of boardwalks to facilitate viewing and sampling of the wetlands. Since the degree of contamination from groundwater inputs and/or seepage from dyke drainage into this wetlands is not known, experimental data are preliminary and not cited herein.

Constructed Wetlands Research Facility

To provide a system adequate for the evaluation of two major wastewater types, Suncor constructed a series of nine replicate, parallel trenches (50 × 3.5 m) with the following characteristics: mean slope of 0.5%; side wall slope of 2.5:1; lining with an impermeable 40 mil high density polyethylene liner; a clay liner topped with rock rip rap at the outflow, with a liner-covered wooden dam, a broad-crested weir at the outlet, and piping at the inlets for control of flow [10]. The nine trenches were planted with vegetation (*Typha latifolia* or cattails and *Scirpus validus* or bulrushes) transplanted from two donor sites on the Suncor lease.

5.1.2 Wetlands Function

The concept of using a wetlands only as a polishing system reflects the long retention periods that appear to be required for constructed wetlands when used as primary treatment systems for dyke drainage water. As a polishing system, wetlands would receive water previously treated; for example, in water capped fine tails lakes or in primary treatment ponds. However, Consolidated Tails release water may be more easily treated than dyke drainage water [23]; therefore, it may be possible to utilize wetlands as primary treatment systems for CT water.

Treatment Performance

Optimum contaminant loading rates and HRTs could not be confidently predicted from empirical data for Suncor's dyke drainage wetlands; that is, a prediction of loading rates and HRTs that would both reduce contaminant concentrations in outflow streams to acceptable levels and eliminate toxicity. Estimates of the HRT required to reduce toxicity to between 50 and 100% of control values (*i.e.*, control wetlands using surface runoff water) varied from 11 to >100 d [19]. Although, a consistent trend in contaminant and toxicity reduction was frequently observed with HRTs from 0 to 24 d, continued improvement in water quality (*i.e.*, treatment performance) at 72 d was minimal or non-existent. The inability of empirical data to allow a confident prediction of the optimum HRT required for successful treatment was based on the difficulty in building large pilot-scale wetlands systems that could provide very long retention periods.

Recommended HRT values for the treatment of dyke drainage water in wetlands were established using a criterion of 50% of toxicological or chemical values achieved with control wetlands. For example, if there was 60% survival of a specific organism in control wetlands, then the goal was to achieve 30% survival in the dyke drainage treatment wetlands. The 50% level was chosen since complete detoxification rarely occurred in the treatment wetlands and extrapolation of these data to 100% could not be justified. In terms of removing 50% of the toxicity, HRT values ranged from 11 d to >100 d. For chemical parameters at the 50% level, HRT values were >4 d for ammonia and >60 d for TEH (total extractable hydrocarbons). To achieve 100% of control water quality in terms of rainbow trout survival (a standard regulatory test which generally requires 100% survival compared with controls), an HRT of 45 d was required.

Factors limiting treatment performance likely included: short circuiting/stagnation; low/limiting dissolved oxygen (due to limited mixing); and low levels of nutrients (*i.e.*, phosphates). For this reason, a managed wetlands approach has been recommended. Additions of oxygen and nutrients can address potential limitations of the treatment system. Some preliminary work has shown that oxygen or phosphate addition can enhance wetlands treatment effectiveness. Extensive bench-scale work confirms this conclusion [24]. Therefore, successful treatment could be achieved more quickly using a managed wetlands approach, and 1994 fish toxicity data have confirmed this conclusion [31]. When phosphate was added to inflow water, the HRT required to achieve a level of toxicity to fish for dyke drainage effluent water that would be comparable with control water (*e.g.*, criteria goal of 100%) was reduced from 45 d to <28 d and further reductions are likely since this aspect of treatment has not be fully investigated in field-scale trials.

Finally, although toxicological criteria do not exist to regulate the discharge of treated process-affected water, some chronic (*i.e.*, long-term) reproductive or survival tests will likely be required. For example, pulp mills are now required to test their effluent using similar chronic or reproductive tests under the federal EEM (Environmental Effects Monitoring) program.

Ecosystem Diversity

Plankton communities (*i.e.*, algae and zooplankton) in constructed wetlands used to treat oil sands wastewater will inevitably be different from "natural" communities in nearby wetlands due to inputs of organic and inorganic compounds which act as food for these organisms, either directly or indirectly, and which do not occur in natural systems. In the existing Suncor wetlands facility, there were no differences in benthic invertebrate communities, possibly because even the control wetlands were too small to support a "natural" community (*i.e.*, similar to reference wetlands). Communities of zooplankton in the control wetlands were less similar to natural communities than were communities of the smaller phytoplankton, again suggesting scale limitations. Differences in the ecology of constructed wetlands used to treat dyke drainage compared with wetlands with inputs of only surface runoff water consisted of: reduced species diversity for zooplankton and phytoplankton or algae; reduced densities of these organisms (although some algal species increased in numbers); and reduced numbers of emergent insects [7,19]. There was no difference in macrophytes (aquatic plants) which were able to thrive in both the control and treatment wetlands.

5.1.3 Active Water Treatment

In engineering terms, wetlands are self-sustaining fixed-film bioreactors [1]. Active treatment (*i.e.*, a managed wetlands) would involve the use of ongoing manipulations to enhance the capability of this bioreactor to treat wastewater.

A conceptualized diagram of the factors which limit treatment processes in a self-sustaining wetlands and the resulting recommendation for a managed or active wetlands is provided in Figure 5-2 [19]. Both phosphate and dissolved oxygen may limit the treatment capability of these wetlands. Under natural or self-sustaining conditions, the microbial nitrification of ammonia may have inhibited the mineralization and subsequent detoxification of organic contaminants (*e.g.*, hydrocarbons, naphthenic acids) by virtue of competition for available phosphate and/or oxygen.

When these two components were supplemented into the wetlands, ammonia concentrations decreased to non-detectable levels. The elimination of ammonia would, in turn, allow more oxygen and phosphate to be utilized by bacteria for the mineralization of organic contaminants. Therefore, the reduction of ammonia could be undertaken as a pretreatment step and might be achieved in aerated lagoons/wetlands or holding ponds. Final treatment of the wastewater for the removal of more persistent organic contaminants could then likely be accomplished in a conventional constructed wetlands.

5.2 Environmental Considerations

The discharge of contaminants into a constructed wetlands would likely be more acceptable to regulatory agencies compared with discharge into a pre-existing natural wetlands, which would likely be altered in some manner or suffer some adverse ecological impacts [25]. However, adverse ecological impacts even to constructed wetlands are clearly undesirable. Therefore, effective constructed wetlands treatment systems should incorporate a sustainable, healthy biological community [26] although this community would not be the same as nearby natural communities.

5.2.1 Assimilative Capacity

The capacity of a wetlands to assimilate or biodegrade organic contaminants into carbon dioxide (kinetic design criteria) has been based on "corrected" mineralization rates to neutralize any impact of natural organic compounds. These rates are based on the difference in rates of carbon dioxide production between Dyke Drainage wetlands and Control wetlands. This corrected value was considered a reflection of added inputs of organic contaminants, most notably naphthenic acids and TEH [27]. Values for TEH mineralization need to be confirmed using suitable marker compounds, that is, by specific analysis using either chemical methods (most organic compounds in dyke drainage water are not readily identifiable at present and the measurement of TEH is

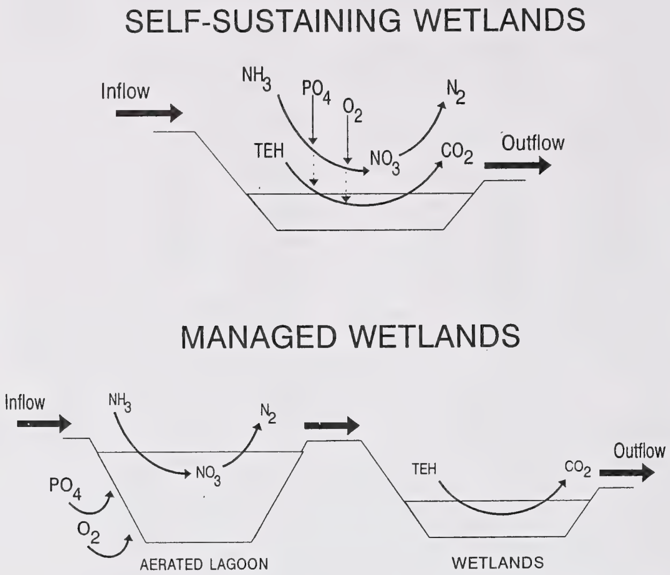

FIGURE 5-2. Conceptual designs for both self-sustaining and managed wetlands showing the advantages of phosphate addition and aeration [19].

considered a gross indicator of contamination) or through the use of radio-labelled surrogate compounds (these experiments are ongoing). However, the estimated "TEH" mineralization rate in the treatment wetlands likely reflects the removal of organic contaminants since it has been corrected by subtracting mineralization rates from control wetlands and since the input of nutrients with these contaminants did not appear to stimulate the biodegradation of natural organic compounds such as leaf litter [7]. Combining 1993 and 1994 data, the average estimated TEH mineralization rate for dyke drainage wetlands was 11 mgC/m^2/h [19].

The similarity in corrected mineralization values for the two years (11.4 and 10.5 mgC/m^2/h), notwithstanding a substantial decrease in inflow rates (*i.e.*, contaminant loading) from 6 L/min in 1993 to 2 L/min in 1994, suggests that the capability of microbial mineralization processes in these treatment wetlands is fixed regardless of the level of contaminant inputs. This fixed rate of mineralization is likely due to limitations caused by a number of factors. Four major environmental factors (phosphorus, DO, surface area, temperature) appear to play a role in limiting the rate of mineralization in Suncor's constructed wetlands.

The presence of one or more limiting factors is also suggested by the relatively low rates of mineralization in these wetlands compared with both control wetlands and values in the literature [7]. The rate of removal for conventional RBCs (rotating biological contactor) was 36 to 78 mgC/m^2/h in the treatment of oily wastes [28]. These values are substantially higher than the 11 mgC/m^2/h reported here and likely reflect the increased surface area provided by these engineered (biofilm) systems and the relatively low level of organic contaminants present in oil sands wastewater. A mineralization rate of 11 mgC/m^2/h converts to 96 gC/m^2 per year, which is low compared with values obtained from the scientific literature. For example, values have been reported ranging from 254 to 804 gC/m^2 per year for total carbon dioxide release rates from a salt marsh and lake sediment, respectively [29]. In a study of constructed wetlands, mineralization rates were found between 361 and 733 gC/m^2 per year [30].

The contaminant loading rate in each replicate dyke drainage wetlands (59 gC/m^2 per four months) was about twice the mineralization rate (31 gC/m^2 per four months). Therefore, using these calculations to derive preliminary estimates of treatment capability, an HRT of about two years or 240 days (*i.e.*, two summer of four months each) would be required as a holding time for the complete mineralization and treatment of dyke drainage water. The 240 d retention time was calculated at a flow rate of 2 L/min, a TEH concentration of 35 mg/L (1994 mean), and using a land area of 0.0175 ha (*i.e.*, existing trenches). As a rough estimate, assuming microbiological processes increase with increasing surface in a linear fashion, a Dyke Drainage flow rate of 1,000 L/min would require 9 ha for treatment; and 10,000 L/min would require 90 ha over two years.

5.2.2 Contaminant Fate

Effluent quality in the Suncor wetlands was assessed by comparing water outflow quality from Control and Dyke Drainage wetlands to ambient water quality and provincial and federal guidelines (Table 5-1). Dyke Drainage water quality exceeded regulatory guidelines for several water quality parameters. However, outflow water quality was often comparable or better than that observed in nearby water bodies (*e.g.*, Athabasca River, Ruth Lake) for the same water quality parameters that exceeded guidelines. For example, iron and manganese guidelines were almost always exceeded, although iron and manganese in the Dyke Drainage outflows were similar to levels found in the Athabasca River and Ruth Lake. In addition, the discharge from control wetlands using nearby surface runoff water also exceeded some guidelines (*e.g.*, iron), but not background levels.

A toxicity identification evaluation (TIE) study [31] showed that there were three potential classes of toxic materials present in dyke seepage water: ammonia, naphthenic acids, and phenolic substances. Ammonia was largely responsible for toxicity associated with fathead minnows. The results of this study also indicated that wetlands were able to treat all organic toxicants equally; that is, there was a general reduction in levels with passage through the wetlands.

The fate of specific contaminants, or groups of contaminants, is discussed as follows.

Hydrocarbons (Total Extractable Hydrocarbons or TEH)

Naphthenic acids are included as a subcomponent of TEH since the analytical technique used to extract and quantify TEH compounds has been shown to include this class of organic acids [32]. TEH removal rates for dyke drainage water on a concentration basis were generally low (6 to 24%). However, low removal rates can be partly attributed to the offsetting impact of concentration effects due to evapotranspiration. The total estimated load of TEH removed was much higher (22 to 79%) suggesting that constructed wetlands can remove substantial amounts of TEH [19]. There was no indication that TEH compounds were accumulating in sediments.

The majority of TEH removal in Suncor's constructed wetlands was accomplished through processes of bacterial mineralization [19]. Average mineralization rates (31 g C/m^2/4 months) were comparable to observed removal rates (ranging 11 to 47 g C/m^2 per four months, and averaging 24 g C/m^2 per four months). The lack of significant TEH accumulations in the sediments and the similarities between rates of mineralization and removal suggest that wetlands treatment of hydrocarbons is primarily microbiological.

Most TEHs in Suncor's oil sand wastewater occur in the C_{15} to C_{28} range. Chromatograms of the TEH extracts indicate that the wetlands effectively removed many hydrocarbons in this range as well as those hydrocarbons which occur sporadically in the C_9 to C_{15} range [32].

Notwithstanding that mineralization is the major pathway for hydrocarbon removal, bioaccumulation of TEH compounds within insect larvae can occur [7]. Concentrations of organic compounds (*i.e.*, no positive identification) were found at concentrations ranging from 5 to 10 ppm; however, impact on these organisms or the extent of transfer up the food chain are not known.

Preliminary analysis of TEH within cattail and bulrush roots suggests that oil sands hydrocarbons are not readily accumulated by plant tissues [7]. However, a weak trend for TEH within cattail root and wetlands TEH loading rates was noted in the Suncor constructed wetlands; that is a loading of 10 kg TEH per wetlands within a four-month period resulted in the accumulation of 89 grams of TEH within cattail roots.

Naphthenic Acids

A technique developed by Syncrude Canada Ltd. has enabled specific analysis for these organic acids. Naphthenic acids are a mixture of water soluble mono- and polycyclic alkanes with side chain bearing a carboxyl group and are a principal toxic component in tailings pond water [16,33]. Since naphthenic acids are partially biodegradable [34], bacterial degradation processes in wetlands should be capable of reducing the toxicity of oil sands wastewater. In biometer microcosms (*i.e.*, a small flask with wetlands sediment, water, and inoculated with naphthenic acids), toxicity was reduced in Suncor's wastewater by approximately 30 to 40% [32], indicating that the biodegradable and toxic portion of naphthenic acid may be quickly mineralized by bacteria in a simulated wetlands environment. Naphthenate biodegradation, by aerobic bacteria, is largely responsible for the detoxification of tails pond water in Syncrude's experimental pits [33].

Ongoing research is exploring the fate of radiolabelled naphthenic acids in both laboratory biometers and field experiments.

TABLE 5-1. Effluent quality of the outflow waters from Suncor's constructed wetlands compared with ambient water quality in the Athabasca River and Ruth Lake and with Alberta Environmental Protection's Surface Water Quality Objectives (ASWQO) and with Environment Canada's Canadian Council of Resource and Environments Ministers (CCREM) Water Quality Guidelines.

Parameters	Constructed wetlands outflow water						Athabasca River	Ruth Lake	Alberta ASWQO	Environ. Canada CCREM
	Control		Dyke drainage-rep		Dyke drainage-split					
	min	max	min	max	min	max				
WATER QUALITY										
Conductivity (μS/cm)	456	733	969	1792	1241	1894	292	360	–	–
Dissolved Oxygen — DO (mg/L)	3.4	13.8	3.8	11.1	1.2	10	1.7–10	8.4	>5	>5
pH	7.4	8.5	7.6	8.4	7.6	8.3	8.2	8.3	6.5–8.5	6.5–9.0
Turbidity (NTU)	0	59	4	107	0	122	3	25	–	
NUTRIENTS (mg/L)										
Total Kjeldhal Nitrogen — TKN	0.24	1.22	2.64	7	0.36	14.3	0.7	–	–	–
Ammonia — NH_3	0.001	0.076	0.001	10.86	0.001	14.6	0.09	0.2	–	1.37
Total Phosphorous — TP	0.029	0.061	0.116	0.17	0.08	0.08	0.48	–	0.05	–
ORGANICS (mg/L)										
Total Extractable Hydrocarbons — TEH	1	2.6	10.7	38.7	17.5	47.4	–	–	–	–
Total Organic Carbon — TOC	7	23	37	71	40	68	–	22	–	–
OXYGEN DEMAND (mg/L)										
Biological Oxygen Demand — BOD	5	5	5	5	5	10	–	3	–	–
Chemical Oxygen Demand — COD	32	83	182	341	194	335	–	66	–	–
MAJOR IONS (mg/L)										
Potassium — K	4	6.7	14.8	16.7	13.8		1.4	3.7	–	–
Calcium — Ca	51.9	84.2	34.7	35.5	30.4	44.8	40	–	–	–
Magnesium — Mg	20.4	22.5	16	16.8	14.4	17.8	11	16	–	–
Sodium — Na	63	73	380	391	341	399	15	33	–	–
Chloride — Cl	46.8	50	33.9	36.5	32.3	34	12	7.2	–	–
Sulphate — SO_4	36.9	69.6	114.8	130.5	137	163	147	10	–	–
METALS (mg/L)										
Aluminum — Al	0.2	0.31	0.3	0.47	0.2	0.56	0.27	–	–	0.1
Barium — Ba	0.071	0.095	0.09	0.112	0.081	0.109	0.08	–	1	–
Chromium — Cr	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.006	0.1	0.02
Copper — Cu	0.01	0.01	0.01	0.01	0.01	0.01	–	–	–	0.002
Iron — Fe	0.349	0.778	0.85	1.75	0.334	1.2	0.8	0.2	0.3	0.3
Manganese — Mn	0.049	0.102	0.049	0.139	0.074	0.137	0.4	0.015	0.05	0.1
Mercury — Hg (μg/L)	0.05	0.05	0.05	0.05	0.05	0.05	0.1	<0.5	0.1	0.1
Molybdenum — Mo	0.03	0.03	0.03	0.03	0.04	0.07	0.1	–	–	–
Nickel — Ni	0.02	0.02	0.02	0.02	0.02	0.02	0.02			0.025
Strontium — Sr	0.206	0.241	0.484	0.541	0.498	0.643	0.24	–	–	–
Vanadium — V	0.03	0.03	0.03	0.03	0.03	0.03	0.001	–	–	–
Zinc — Zn	0.005	0.013	0.006	0.019	0.007	0.011	0.008	0.012	0.05	0.3

Metals

Concentrations of all heavy metals (*e.g.*, iron and aluminum), although variable, decreased in Suncor's constructed wetlands from inflow to outflow [32]. Heavy metals are likely closely associated with the suspended particulate fraction of water. As these particulates settled, metal concentrations in the water column were correspondingly reduced.

Metals were generally not taken up into plant tissue [7]. The exception to this was elevated concentrations of aluminum in both cattail and bulrush shoots, which tended to be greater in those plants sampled from Pond 1A wetlands *versus* control wetlands.

Most metals did not appear to be bioconcentrated in wetlands insect larvae. However, lead was an exception. In 1993, both larval chironomids and emergent insects from treatment wetlands (using pond 1A water) had higher concentrations of lead than in insects sampled from Control wetlands [7]. This phenomena was not observed in subsequent monitoring in 1994 and therefore needs to be confirmed [19].

Ammonia-Ammonium

Ammonia is discharged to tailings ponds/settling basins *via* process waters from the refineries. Ammonia-oxidizing bacteria (nitrifiers) likely convert it to nitrate in oxygenated surface waters of a pond or wetlands. In addition, volatilization can occur. Therefore, ammonia could be considered a transient contaminant; indeed, it has been observed to be depleted rapidly from the surface water of an inpit pond [16]. However, since ammonia levels cannot completely account for the toxicity of dyke drainage water to rainbow trout [19], other toxic contaminants (*e.g.*, naphthenic acids, TEH) must be present in oil sands wastewater.

Ammonia-ammonium nitrogen removal rates in Suncor's constructed wetlands on a concentration and load basis were high and ranged from a low of 44% to a high of 97%. These removal rates fall in the range reported in the literature; for example, in a literature review, removal rates ranged from 50 to 97% [35]. In addition, the observed ammonia removals were consistent with those predicted by wetlands nitrification modelling, based on the available surface area, hydraulic retention times, and liquid temperatures. The addition of phosphorus to wetlands trenches treating dyke drainage water resulted in enhanced ammonia removal above that predicted by the model [23].

Conductivity

The present conductivity of oil sands wastewater is about 1700 μS/cm compared with Ruth Lake values of 360 μS/cm or Athabasca River water of 280 μS/cm [16] — about a five-fold increase. In terms of salinity, tailings top water values are about 1,000 mg/L. Since most freshwater organisms can tolerate levels up to 6,000 mg/L [36], any input of this water into nearby water bodies would not substantially alter the possibility for colonization by most indigenous biota [37].

The Suncor constructed wetlands did not remove most dissolved solids; a modest decrease in overall concentrations was the result of the removal of the sulphate ion — likely through microbial reduction processes to produce hydrogen sulphide [32]. For other ions such as sodium and chloride, an increase in concentrations in wetlands can be predicted as a result of evapotranspiration. The mean evapotranspiration rate was shown to be 2.93 mm/d [19].

Increasing concentrations of cations may influence the number and type of aquatic species that can colonize wetlands water. In Ruth Lake, the major cations in order of dominance are Na>Ca>Mg>K [38]. However, in oil sands wastewater, Na is the dominant ion by a factor of five to ten times the level in Ruth Lake. This high sodium level may limit species diversity for zooplankton or phytoplankton taxa which can successfully colonize wetlands waters. Indeed, a reduction in diversity has been confirmed in the field although the precise cause was not identified [7,19]. However, total plankton abundance or productivity may still approach natural levels [36].

5.3 Long Term Sustainability

A preliminary study showed that a treatment wetlands has the potential to provide a passive, low-cost, self-sustaining system which could detoxify oil sands wastewater [11]. In addition, wetlands would likely contribute valuable ecological benefits to the reclaimed lease area (*e.g.*, plant and animal productivity and diversity, waterfowl habitat). Therefore, the overall reclamation plan envisions a landscape comprised of about 50% forest, 20% grasslands, and 30% lakes and wetlands [12].

5.3.1 Modelling

Wetlands research has consisted of bench-scale and field-scale trials; further contaminant and hydrological modelling may aid in the scale-up of these wetlands into operational units.

5.3.2 Ecosystem Development

Oil sands wastewater treatment wetlands will be colonized by a wide diversity of aquatic plant and animal life and will function as a viable ecosystem. Depending on the type of hydrocarbon and/or other contaminant inputs, the aquatic food chain will differ somewhat from nearby natural systems. For example, different algal species may predominate and certain species of aquatic invertebrates may not thrive in a wetlands treatment system for oil sands wastewater [19]. Some accumulation of contaminants in the food chain may also occur, but it is unlikely that this would constitute a substantial threat to the wetlands or adjacent ecosystems. Further research is required to confirm this latter conclusion.

The status of current wetlands ecosystem development plans and directions for future research are summarized through a series of expanding ecosystem zones or "boxes" (Figure 5-3). In Ecosystem #1, the treatment of tails is conceptualized with the introduction of contaminants (*e.g.*, hydrocarbons, ammonia-ammonium) and dissolved ions (*e.g.*, Na, Ca) into the wastewater stream. Ecosystem #2 envisions a holding pond/shallow lagoon with a defined hydraulic retention time and which would have an effluent discharge criterion of no acute toxicity to fish and other environmental standards to minimize any toxicity to birds or wildlife (perhaps similar to the status of existing tailings ponds). Treatment performance could be enhanced through the addition of phosphorus, increased aeration, and, perhaps, by seeding acclimated bacterial sludge back into the pond from a sidestream treatment area.

Ecosystem #3 summarizes current estimates of the capabilities of a managed wetlands. The main goal of this wetlands area would be to: (1) remove any chronic toxicity from the water and (2) achieve an effluent water chemistry that meets regulatory criteria or is environmentally acceptable with regard to contaminant levels in nearby water bodies. This conceptualized wetlands would be a water treatment system and therefore neither should, nor could be expected to have ecological characteristics which are similar to nearby natural wetlands areas. Within this area, small ponds near the outflow would allow biomonitoring surveys to confirm the environmental acceptability of this water before its discharge into the receiving environment.

Discharge from the wetlands area into an on-site lake (Ecosystem #4) would provide a test area which would allow biological monitoring and chemical testing to satisfy any concerns from the public or regulatory agencies regarding the

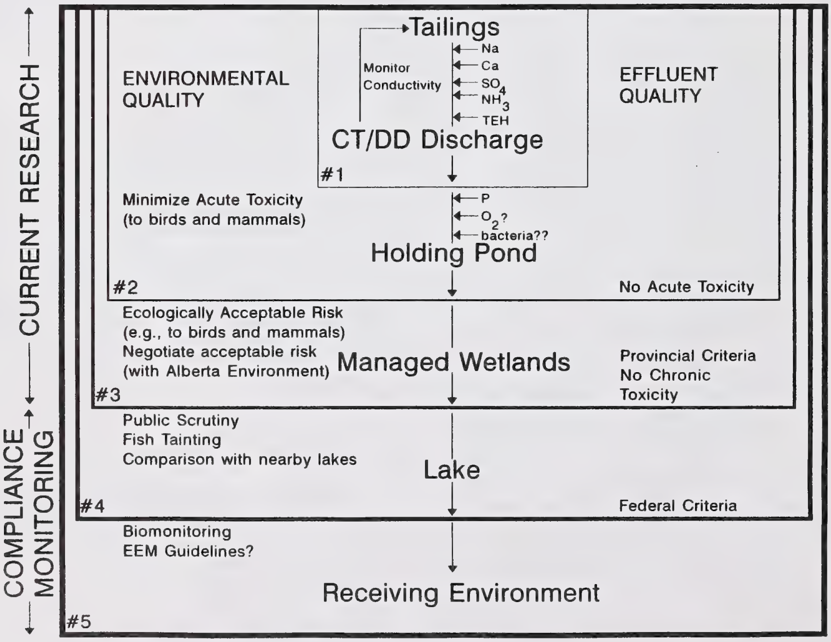

FIGURE 5-3. An overview of environmental, research, and regulatory considerations involved in the various components of a scenario to treat oil sands wastewater [19].

possible impact of treated wastewater on the aquatic environment (Ecosystem #5). This could be done by ongoing biomonitoring surveys or special research projects. Final discharge into Ecosystem #5 (*e.g.*, Athabasca River) would need to meet both provincial and federal water quality criteria.

5.3.3 End Use

Wetlands areas may function primarily as a safeguard or buffer zone between the reclaimed areas and the off-lease receiving aquatic environment [9]. These workers suggested that the main concerns identified for oil sands wastewater would be related to both the large volumes available for release and the acceptability of various reclamation landforms, rather than water quality. Since the quality of water flowing into any treatment wetlands is expected to be good, these wetlands should have a high degree of public acceptability since they would add important ecological benefits to the total landscape (*i.e.*, terrestrial and aquatic) such as increased biological diversity [1].

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6.0 RECLAMATION LANDSCAPE MODEL

The Reclamation Landscape Model Development Project was initiated in 1991 by Suncor and Syncrude to develop a modelling framework to assist in reclamation planning. The initial year of model development focused on developing a suite of computer simulation models suitable for evaluating hydrodynamic, water quality, and biological conditions of fine-tails bottom lakes that would be capped with a layer of fresh water, *i.e.*, wet landscape reclamation option [1]. That modelling framework (Wet Landscape Module; Section 3.2.3.2) was further refined in 1992–93 by incorporating probabilistic capabilities, developing secondary production and food-chain components, and implementing a model to simulate contaminant concentrations in the Athabasca River [2]. In 1993–94 the emphasis expanded from a focus solely on developing tools for evaluating wet landscape options to developing an integrated modelling framework capable of evaluating both wet and dry landscape reclamation options [3]. Major components completed in 1993–94 included development of a dry landscape module that includes surface runoff, groundwater seepage, air vapor, and bioaccumulation components; development and testing of a wetlands module to assist in evaluating treatment potential and for optimizing design of wetlands units; and development of a risk analysis module for quantifying risks to human health and ecological receptors.

The Reclamation Landscape Model is a compartment modelling system that has been developed to assist in evaluating and optimizing reclamation of oil sands mine sites in northern Alberta. The modelling system consists of a number of stand-alone computer programs that can be run in conjunction with one another or separately. The computer programs that simulate contaminant-fate processes range from simple empirical models to complex, mechanistic, three-dimensional, dynamic models, which are capable of predicting contaminant concentrations in water, soil, air, and biota for a wide range of reclamation landscape units. Most of the component models have probabilistic capabilities, and a Risk Analysis Module utilizes output from these contaminant-fate models to compute risks to human and ecological health.

An overview of the major components of the Reclamation Landscape Model is shown in Figure 6-1. Input data requirements are highly variable depending upon the reclamation landscape to be simulated and the level of detail required. Examples of model input include boundary conditions (spatial and temporal), physical configuration of the landscape unit(s), initial chemical characterization of the waste materials, climatic variables, and physiological and behavioral attributes of receptors (for risk characterization).

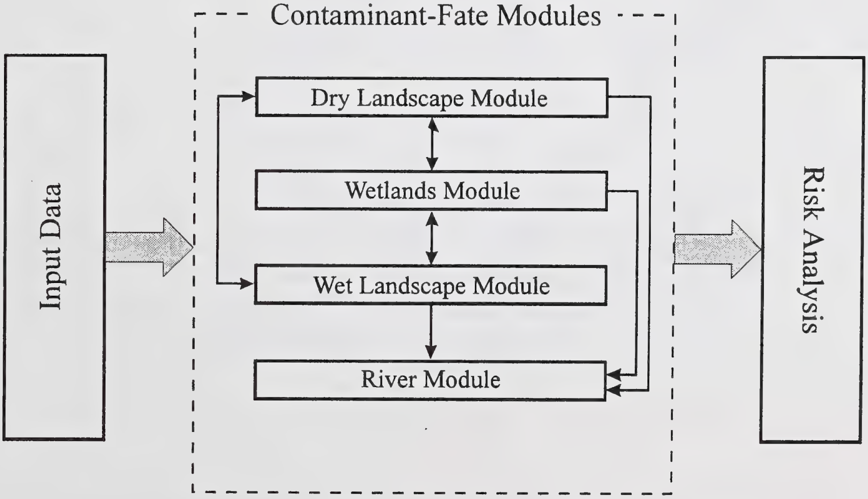

FIGURE 6-1. Conceptual overview of Reclamation Landscape Model [3].

The core of the Reclamation Landscape Model consists of four contaminant-fate simulation modules:

- *Dry Landscape Module* — simulates contaminant concentrations in groundwater, surface runoff, and air, and loading rates to wetlands, water capped fine tails lakes, or streams.
- *Wetlands Module* — simulates treatment efficiency of natural and constructed wetlands.
- *Wet Landscape Module* — simulates contaminant concentrations of sediment, water, and biota in natural lakes and water capped fine tails lakes.
- *River Module* — simulates contaminant concentrations of sediments, water, and biota in the Athabasca River and other streams.

Each of these modules consists of a number of different computer programs (Table 6-1). Selection of a specific program depends upon the level of detail required to analyze a particular scenario. For example, simple analytical programs are appropriate for initial screening of a particular reclamation option. However, the more sophisticated programs may be necessary for more detailed investigations to help identify factors controlling contaminant concentrations in specific environmental media.

The modules may be run independently or in conjunction with one another. Some of the model sub-components are dynamically linked to one another, *e.g.*, the food-chain model FGETS and the water quality model WASP. For other sub-components, output from one model is used as input to another model, *e.g.*, output from the hydrodynamic model CE-QUAL-W2 can be incorporated into WASP input files.

Output from the simulation modules includes predictions of contaminant concentrations in pertinent environmental media. That output can in turn be analyzed with the Risk Analysis Module to quantify exposure doses and risks to human or ecological health. The Risk Analysis Module can be run either deterministically (to give a single hazard quotient, *i.e.*, the ratio of the exposure concentration or dose to the value known to cause an effect) or probabilistically (to quantify the likelihood of adversely affecting the selected receptor).

The Reclamation Landscape Model has already been used by Suncor and Syncrude to assist in evaluating alternative reclamation options and for detailed investigation of specific reclamation materials and landscapes. Table 6-2 lists projects in which components of the Reclamation Landscape Model have been utilized.

TABLE 6-1. Reclamation landscape model computer programs

Computer program	Source	Use
Dry Landscape Module		
Runoff*	Reference [1]	Screening level — Surface runoff volumes and contaminant loads
Seepage*	Reference [1]	Screening level — Seepage concentrations and loads
Air*	Reference [1]	Screening level — Soil and air vapor concentrations
Biota*	Reference [1]	Screening level — Food chain bioaccumulation in plants and animals
Wet Landscape Module		
WASP4*	Reference [4,5]	Detailed simulations — Lake water quality
CE-QUALW2	Reference [6]	Detailed simulations — Lake hydrodynamics and thermal structure
FGETS	Reference [7]	Detailed simulations — Aquatic food chain accumulation
LAKE*	Reference [2]	Screening level — Lake water quality and food chain bioaccumulation
SECOND	Reference [5]	Screening level — Secondary production
Wetlands Module		
WASP4*	Reference [1]	Wetlands treatment potential
River Module		
WASP4*	Reference [2,4]	Detailed simulations — River water quality
FGETS	Reference [7]	Detailed simulations — Aquatic food chain accumulation
RIVER*	Reference [3]	Screening level — Water quality and food chain bioaccumulation

* Probabilistic capabilities have been developed.

TABLE 6-2. Applications of reclamation landscape model.

Project	Components used	Notes
Syncrude ERCB Hearings [2]	Wet Landscape Module Athabasca River Module	Long-term simulation of contaminant concentrations in water capped fine tails lakes and the Athabasca River
Oil Sands Performance Assessment [8]	Dry Landscape Module Wet Landscape Module Athabasca River Module	Risk Analysis Module Evaluation of chronic release from alternative, hypothetical reclamation landscapes
Dry Landscape Risk Assessment [9]	Dry Landscape Module Risk Analysis Module	Quantity risks associated with alternative dry landscape and reclamation materials
Tar Island Dyke Risk Assessment [10]	Wet Landscape Module Athabasca River Module Risk Analysis Module	Quantify risks associated with seepage from TID

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7.0 SUMMARY AND RECOMMENDATIONS

7.1 Water Capped Fine Tails Lakes Summary

The wet landscape option leaves the fine tails as a fluid, which means that they are non-trafficable and require containment in geotechnically secure areas. Conceptually, the fine tails capping option disposes of mature fine tails as a fluid (>30 wt% fines), over which a layer of water is placed. Containment within the mine pits would be below original grade and depths of more than 40 m of fine tails are expected with a water capping layer of 5 m, to form the fresh water lake. Investigations have indicated that a capping depth of 5 m would be sufficient to prevent resuspension during a one in one hundred year storm for a lake with a fetch of 4 km.

Within one to two years of capping, it is anticipated that acute toxicity to fish and other biota would disappear. Over time, a detrital layer will build up over the fine tails zone, serving both as a barrier to potential mixing of the fine tails into the capping water, as well as a biologically-active medium to enhance degradation and mineralization of compounds released by the fine tails.

Fates of oil sands related components associated with expressed fine tails porewaters generally fall into three groups: (1) soluble organics that are volatile and/or easily biodegradable, (2) compounds that are oxidized (*e.g.*, ammonia), and (3) compounds that adsorb onto particulate matter and/or precipitate as a result of changes in redox conditions (*e.g.*, metals).

A modelling framework has been developed to assist in planning for oil sands mine reclamation. This Reclamation Landscape Model includes a Wet Landscape Module which can be used to predict water and sediment concentrations of the key inorganic and organic variables. In addition, concentrations of organic contaminants in aquatic biota (including fish) can be predicted. The computer programs simulate contaminant-fate processes ranging from simple empirical models to complex, mechanistic, three-dimensional, dynamic models, which are capable of predicting contaminant concentrations in water, soil, air and biota for a wide range of reclamation landscape units. The Reclamation Landscape Model has has been calibrated to and tested against water quality data collected from Syncrude and Suncor's experimental Pits.

7.2 Dry Landscape Summary

Dry landscape scenarios have two principal environmental concerns: the impact of any released water on the aquatic and/or terrestrial environment; and, the characteristics of the solid material as a solid deposit. In the past years, several concepts for dry land reclamation have been tested in both laboratory and field studies (*e.g.*, freeze-thaw, natural evaporation). In most cases, the experimental focus was to assess the capability of a particular technique to dewater fine tails and to produce a solid deposit. The new focus has been placed on the quality of water released, fate of contaminants, or long-term sustainability in terms of environmental impacts.

In terms of the environmental characteristics of the dry tails (*i.e.*, potential soil material), the most comprehensive study to-date has demonstrated variable toxicities, ranging from low to high, for various combinations of fine tails with other materials. Therefore, further investigations are needed if fine tails material is to be disposed of within the biologically active layer of the soil. One area which received more attention in terms of environmental impacts has been the concept of using fine tails as a soil amendment, a technique which is not practicable in terms of the disposal of more than a small fraction of the available fine tails. More likely, however, dry tails material will be buried too deep to be of concern in terms of most potential environmental impacts, and the principal remaining concerns will be the pathways, rate, quality and impact of any waters released from the deposits.

7.3 Wetlands Summary

Research has provided preliminary design criteria, that may ultimately be used for the construction of wetlands to successfully treat oil sands dyke drainage water at optimum loading rates. These experimental wetlands demonstrated that under specific loading rates, an effluent quality acceptable for discharge into the receiving environment could be achieved [7]. This conclusion was based on both regulatory standards as well as comparisons with the water quality of nearby lakes and rivers.

Kinetic analysis (*i.e.*, bacterial mineralization rates) can provide a method to estimate the size of a wetlands required to treat seepage water with a known extractable hydrocarbon concentration and at a specific flow rate (*i.e.*, loading rate). However, further research is required to confirm these design criteria since the characteristics of treatment processes within these wetlands are not completely understood.

A viable ecological community was established in pilot-scale treatment wetlands at the Suncor site. For example, the accumulation of hydrocarbons into sediments, plant and/or insect tissue was minimal and transitory and indigenous communities of phytoplankton, zooplankton and aquatic plants were abundant.The use of constructed wetlands to treat seepage water can provide not only a low cost, low maintenance treatment system, but also can provide increased amounts of wildlife and waterfowl habitat to the overall landscape.

7.4 Recommendations

Fine tails capping as a concept appears technically possible. Monitoring of the large scale capping demonstration, at Syncrude site, should continue.

The passage of seepage water through a wetlands into the surrounding environment is potentially an integral component of fine tails reclamation, whether it be wet or dry. Research to understand better the use of wetlands and the resulting water quality should continue through the application of field scale demonstrations.

Although there has been considerable effort aimed at field pilot testing the feasibility of specific dry landscape options, the focus of the more fundamental reclamation research (issues such as contaminant release and fate) has dealt more with the water capped lakes and wetlands options. Consequently, more of this type of fundamental research into the dry landscape options is essential, particularly with regard to contaminant fate issues and long term stability of a "dry tails" deposit. In addition, research into the benefits of using fine tails as a soil amendment should also continue.

The chronic toxicity testing of fine tails that has occurred to date has not provided any firm conclusions. Research should continue into the development of chronic toxicity tests which could address the specific concerns of all stakeholders.

INDEX

VOLUME III

VOLUME REDUCTION OF CLARK HOT WATER EXTRACTION FINE TAILINGS

CONTENTS

INTRODUCTION TO VOLUME III

This Volume of the Fine Tailings Fundamentals Consortium Final Report focuses on reduction or elimination of the accumulation of fine tailings produced by the Clark Hot Water Extraction (CHWE) process, which is the process currently employed by the two commercial operations in Alberta. Volume I indicated that approximately 500 million cubic meters of fine tailings are now impounded at the two commercial operations and discussed the extremely slow settling behavior and generally intractable nature and properties of fine tailings. Volume II describes the nature of organic contaminants contained within tailings pore waters and the associated environmental impacts as well as reclamation alternatives employing water capped lakes and wetlands treatment of effluent water. Volume IV covers alternative extraction processes that may potentially significantly reduce or eliminate fine tailings formation or accumulation.

The search for economic and environmentally acceptable methods to reduce the accumulation of fine tailings has been underway for more than two decades. There are two major incentives for this effort. First, there are significant operating costs associated with providing storage for fluid fine tailings and with the various pumping activities associated with movement between short and long term containment structures. Second, there is a risk with long term storage of fine tails that loss of containment structures might lead to a future environmental discharge. The list of methods investigated by a large number of investigators covers nearly every conceivable approach with testing ranging from laboratory experiments through to large pilot test programs. None of those investigations produced results which met the various criteria established for commercial implementation.

The Fine Tails Fundamentals Consortium was established to develop an understanding of the nature of fine tailings and to use that fundamental understanding to propose and test practical methods to improve fine tailings behavior. This volume summarizes the fundamental understanding gained on approaches which have led to alteration of CHWE fine tailings behavior. Data describing the altered properties are presented. The ability to alter fine tailings behavior has recently led to large scale pilot testing of two technologies: (a) natural freeze-thaw processing of chemically treated fine tailings and (b) chemical treatment leading to incorporation of the fine tailings in a nonsegregating mixture of plant tailings which consolidates relatively quickly in tailings deposits. The significance of these tests was that they were successful on a large scale, and that they were the result of collaboration among the members of the Consortium. In both cases previous work had been done by others, but it is only now that sufficient understanding of the process fundamentals, commercial application opportunities, and economic evaluations have established the feasibility of the processes in a commercial environment.

Any attempt to reduce the volume of fine tailings and convert it to a deposit with soft soil-like consistency will result in the release of water currently stored within the fine tailings. There is little advantage to either the operating companies or the public if this water must be stored at the mine site. There are two ways to deal with this water. If it possesses suitable chemical compatibility with the extraction process such that bitumen recovery is not degraded, then it can be used for process make up water as discussed in Chapter 5.0. If not, then it can be treated to remove any sensitive contaminants and discharged to the environment as discussed in Chapter 6.0.

1.0 OPPORTUNITIES FOR FINE TAILINGS VOLUME REDUCTION AND HISTORICAL REVIEW

1.1 Introduction

The Consortium has clearly confirmed that the volume of fine tailings produced in extraction is directly proportional to the amount of clay fines in the ore that is dispersed during bitumen extraction processing (see Volume I). However, the actual accumulation rate in tailings ponds reflects more complicated relationships. Approximately 10 percent of the clay fines in ore are rejected as clay lumps in screen oversize. The remainder reports to the tailings hydraulic pumping systems. Upon discharge from the tailings pipeline into the tailings pond, the tailings stream separates into a stable coarse sand deposit, and a fluid fines pool. The voids of the coarse sand deposit are filled with a mixture of process water and fines. The remaining fines, the majority of which are clay sized (less than 2 microns), enter the tailings pond, flocculate after an induction period lasting up to several weeks, and then begin the sedimentation and slow dewatering process. After about three years the fines achieve 30 wt% solids, and are said to be mature fine tailings (MFT). During construction of beaches into the tailings ponds, there is a very important process of recapture of mature fine tailings into the beaches. This leaves approximately 32 percent of the original fines contained in the oil sands deposit which accumulate as fluid mature fine tailings.

1.2 Fine Tailings Treatment Opportunities Leading to Volume Reduction

Based on the above discussion, several opportunities exist in the process where reduction of fine tailings accumulation might be accomplished through either mechanical or chemical modifications. In general these opportunities exist at the oil sand mining stage, in the oversize rejection and bitumen extraction stage, after extraction but before disposal to tailings ponds, or after tailings disposal and impoundment. These opportunities are shown schematically in Figure 1-1 and discussed in summary form below.

1.2.1 Selective Mining

There are regions of materials within the ore body containing high clay content and low bitumen content called "interburden." Although it is desirable to selectively mine and reject interburden material, this selectivity was only practical with bucketwheel excavator, conveyor, or dragline, bucket wheel reclaimer, conveyor systems for large interburden deposits. Orderly mine development, and large scale material movement, required that smaller interburden deposits in the ore zone be excavated and sent to the Extraction Plant. The planned conversion by both operators to truck and shovel mining techniques will tend to increase the ability to reject some of the smaller interburden deposits. The reduction of fine tailings accumulation rate is probably less than 5 percent for existing operations.

1.2.2 Alternate Extraction Processes

The proven excellent bitumen recovery efficiency of the existing Clark Hot Water Extraction process over a wide range of ore grades and operating conditions makes it difficult to replace, in spite of drawbacks such as the dispersive nature of the process that contributes to the formation of the MFT. Several proposed alternative extraction processes which potentially achieve economic recovery levels while reducing fine tailings issues are discussed in Volume IV.

1.2.3 Prevention of Segregation of Coarse and Fine Minerals

It is desirable to prevent segregation of coarse and fine minerals such that the fines are incorporated in the sand deposit as they were in the ore body. The process can be significantly enhanced by chemical treatment. Lime addition is an example. Creation of nonsegregating mixtures of plant tailings, and fines, is actively being tested and evaluated as a commercial method of eliminating accumulation of fine tailings.

1.2.4 Recombination of Extraction Tailings and Fine Tailings

Additional fine tailings can be incorporated in the voids of coarse sand deposits by addition of fine tailings to fresh tailings

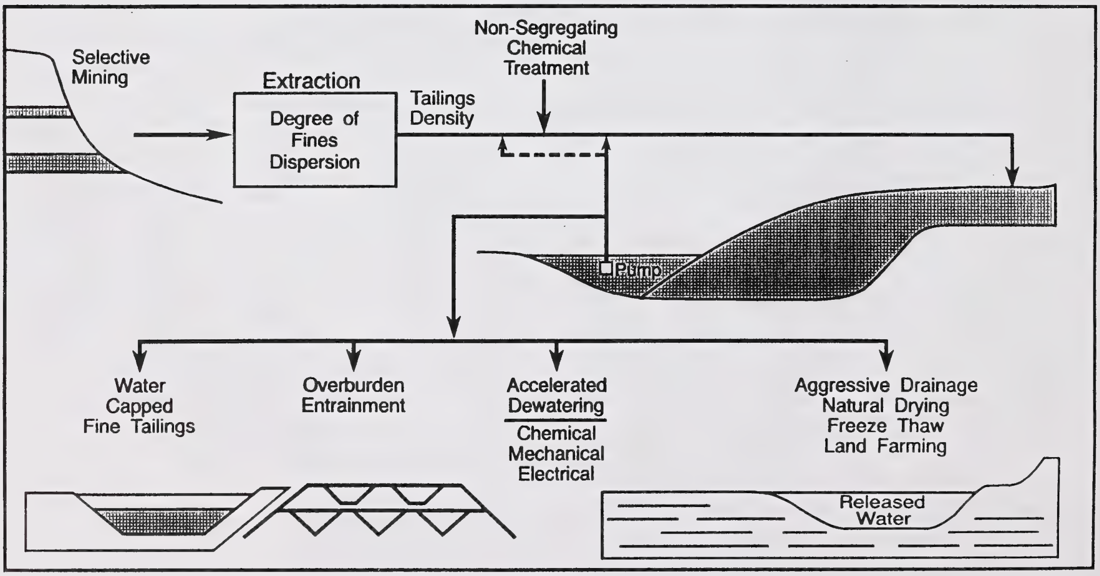

FIGURE 1-1. Fine tailings volume reduction opportunities.

through a process called "spiking" [12]. However, testing has not demonstrated an important recapture process. This approach can be significantly enhanced using a wide range of chemical treatments and mechanical equipment. Creation of nonsegregating mixtures of plant tailings and fines is actively being tested and evaluated as a commercial method of eliminating accumulation of fine tailings. A review of past work and current development is discussed in more detail in Chapter 3.0.

1.2.5 Extraction of Water from Mature Fine Tailings

A wide range of chemical treatments often combined with mechanical equipment [11] has been tested without success at the commercial level. Many of these are presented in the summary in the next section. Natural freeze-thaw of chemically treated fine tailings has been demonstrated on a large scale produce a soil-like deposit from fine tailings. In addition, natural evaporation [10] enhanced by farming techniques have been utilized in many parts of the world to dry dredged materials. On a commercial scale, large areas are required to handle the full accumulation of fine tailings, and time frames in the order of tens of years are necessary to make efficient use of prepared and impounded areas. However the option of application of the process to an existing volume of MFT is available, and is under active appraisal, and possible commercial scale testing.

1.2.6 Stabilization of Mature Fine Tailings

Mature fine tailings can be mixed with other earth materials such that a soft soil is formed thereby eliminating the need for fluid storage, as has been successfully demonstrated by Syncrude. Mixing MFT with the smectitic clay overburden was successfully demonstrated, but will be an option only on leases where sufficient amounts of suitable overburden exist [8,9]. Recent work at the University of Alberta seems to indicate that mature fine tailings can potentially be treated with calcium sulphate to consolidate in a reasonable time frame. This development is discussed in Chapter 7.0.

1.3 Historical Development of Fine Tails Volume Reduction Processes

There are several excellent references that describe work done previously by researchers in this field. What follows is a summary of the work and an explanation of the issues that prevented further development at the time.

- *A Review of Properties and Treatment of Oil Sands Tailings*, AOSTRA Journal of Research, 8(1992)11. by Dr. K.L. Kasperski, is an excellent review of previous work on Fine Tailings Volume reduction efforts.
- *A Catalogue of Technologies for reducing the Environmental Impact of Fine Tails from Oil Sand Processing*, prepared for the Reclamation Research Technical Advisory Committee, and Fine Tails Fundamentals Consortium, covers the state of the art knowledge available at the time of writing.

Tables 1-1 and 1-2 present the information on previous work in summary form. The references provide additional information if required.

Generally these approaches were not practically applicable, because of either high capital costs, high operating costs, or the results were not satisfactory.

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TABLE 1-1. Physical mechanical treatment methods.

Method	Description	References
Centrifugation	With chemical flocculants	16
	Pretreatment with conditioners	17,18
Filtration	Vacuum precoat filtration, pH <7.5, or >9.5	19
	Cross flow microfiltration	20
Electrophoresis	With gravity settling	21
	With pH adjustment to clarify decant	22,23,24
Electrocoagulation		15
Sonic waves	Separate and agglomerate fines, bitumen and water	25
Freeze thaw	various methods, additives	5,13,14,26,27, 28,1

TABLE 1-2. Chemical methods

Method	Description	References
Sodium silicate	CHWE at high clay to water ratios	29,30
Organic flocculants	Acrylic compounds, commercial flocculants, starch, with acids, and others	31,32,33,34,3,7,35,36,37
Inorganic coagulants	Na, Ca, Fe, Al, lime	39,40,4,2,3,41,19, 7, 42,43,44,45,46,47
Altering pH	Improved settling, compaction	3,6,7,16,19,38,40,46,48
Oxidizing and reducing agents	Destroy surfactants	7

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2.0 FUNDAMENTAL BASIS FOR CONTROL OF FINE TAILINGS BEHAVIOR

2.1 Introduction

A model of mature fine tailings (MFT) has been developed based upon the water holding capacity of the mineral components and the floc or aggregate structure of these minerals in suspension. Ultimately, the volume reduction and bulk dewatering behavior of MFT is determined by water holding properties of the clay components.

Clay floc or aggregate structure is known to have a dependence upon both pH and ionic strength of the suspending fluid [1,2]. In the case of oil sands MFT, it has been shown that the clay flocs or aggregates are least aggregated at a pH of about 8.4, determined by the presence of the bicarbonate ion [3]. The clay structure has been observed directly using various microscopic methods and found to be quite different depending upon the pH and presence of various ions [4,5].

Clays, because of their plate-like shape, and differing charges at the surfaces compared to their edges, can become oriented (flocculated or aggregated) in complex ways. Many theoretical treatments have been developed to understand clay behavior in MFT, but basically, there must be some sort of balance between attractive and repulsive forces [6–8]. Attractive components include van der Waals, electrostatic, and hydrophilic forces. Repulsive components include steric and electrostatic. Electrostatic forces can contribute both to flocculation and dispersion in a clay system because the edges and faces can have differing charges: edge to face interactions can be attractive, face to face can be repulsive. At the pH of MFT (8.4), the pH dependent charge on the clay edges is expected to be the same as the faces (negative). Charge quadrupoles (internal to the clay particles) have been proposed as a mechanism for electrostatic attraction between the clay platelets at the pH of MFT [6].

2.2 The Mature Fine Tailings Structure

Work by Kotlyar *et al.* to isolate the hydrophilic mineral fraction of the MFT and demonstrate its gelation behavior is significant evidence in support of the mineral model of MFT water holding capacity [9–11]. Kotlyar determined that the smallest size fractions gelled at the lowest concentrations and that these were consistent with the amounts of these materials in MFT. It has not been established whether or not these "ultrafines" (clays less than 200 nm) are solely responsible for MFT properties. Given the relatively constant surface area to volume ratio for clay platelets as a function of size, and in view of the fractal nature of MFT, the significance of these findings is in the fact that it was the hydrophilic fraction that exhibits these properties.

The Clark MFT structure is characterized by a weakly flocculated clay system that is extremely slow to consolidate. Figure 2–1 illustrates this behavior in a schematic diagram. Fine tailings flocculate with time and settle to form MFT, with its associated properties. The structure of this material is such that there is a barrier to further consolidation. This barrier can be smaller (perhaps nonexistent) with differing extraction processes or with chemical treatments. Or, this barrier can be overcome with mechanical energy input. Figure 2-2 shows a scanning electron micrograph of Clark MFT and the same MFT sample treated with acid and lime. There is a distinct difference in the floc or aggregate structure in these two samples (analogous to the illustration in Figure 2-1). Table 2-1 summarizes these differences (along with a variety of other bulk properties) for several treated samples.

FIGURE 2-1. Schematic representation of MFT formation and the stable structure that is very slow to consolidate. The barrier to change from the stable suspension to one that will eventually consolidate can be lowered by manipulation of the suspension water chemistry or by the input of physical force. The treated Clark MFT (or MFT from non-caustic processes) flocs are stronger and can support a greater stress which will enhance consolidation.

The floc structure set up with Clark MFT is not strong enough to support a load or stress that would begin consolidation. Sand particles simply fall through the weakly flocculated, low strength, Clark MFT. Although a higher

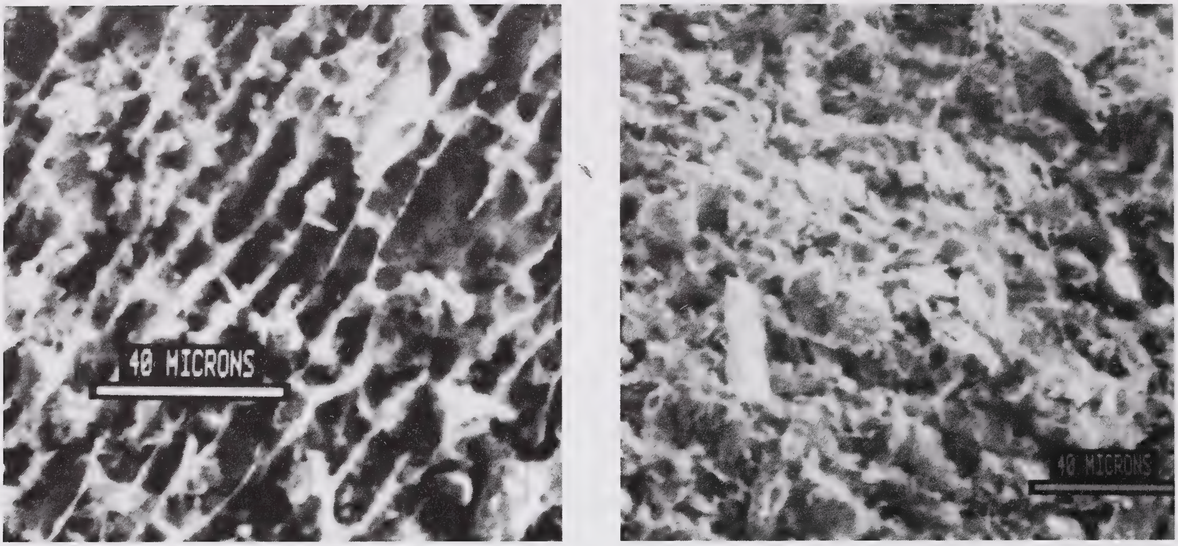

FIGURE 2-2. Electron micrographs of MFT structure before (left) and after acid lime treatment (right). These illustrate the differences in floc or aggregate structure between the Clark MFT and chemically treated MFT.

TABLE 2-1. Microscopic floc structure, rheology, and water release for various MFT treatments. The mean value is the average microscopic (optical) reflectance of the sample which is indicative of the aggregation of the sample. The Clark MFT is the least aggregated of the samples investigated. The structure observed in the scanning electron microscope (SEM) is a subjective assessment of the sample morphology. Examples of the structures referred to are shown in Figure 2-1. The elastic modulus or G' is also a measure of the degree of aggregation. The lower the G', the greater the dispersion of the sample. The specific resistance to filtration (SRF) is another empirical test that is related to the floc or aggregate structure. The higher the SRF, the smaller the flocs or aggregates and the more resistance the sample has to filtration.

Data	Sample identification							
	Clark MFT	OSLO MFT	OSLO MFT + 1500 ppm bicarbonate	HCl to pH 6.0	H_2SO_4 to pH 6.0	HCl to pH 6.0 then $Ca(OH)_2$ to pH 8.0	H_2SO_4 to pH then $Ca(OH)_2$ to pH 8.0	$CaSO_4{\cdot}2H_2O$ 800 ppm Ca
Mean value (x)	2.94	2.00	1.93	2.18	2.63	2.26	2.18	1.87
SEM structure	yes	none	yes	very little	very little	none	none	very little
G'	44	359	179	415	360	402	454	720
% water release (after 1 month)	0			7	8	10	10	10
SRF × 10^{-13} cm/g	6.84	1.2	1.3	1.65	2.63	1.44	.862	.505

solids content material is created, with increasing sand content, the fines to water ratio has not increased, indicating that consolidation is not occurring. By altering the water chemistry of the tailings, stronger floc structures that will support some surcharge are created. In this case, the clay flocs are compressed and the clay to water ratio increases (indicating consolidation is occurring). The different floc structures shown in Figures 2-1 and 2-2 illustrate the changes in floc morphology or structure that occur with changes in water chemistry.

2.3 MFT Floc Structure Modification to Enhance Dewatering

Floc structure and bulk behavior are functions of pH and calcium addition [12–15]. Calcium addition to modify MFT properties can be achieved via acid-lime treatments or by the addition of gypsum. The net effect is the addition of calcium sulphate to the water chemistry although the acid-lime treatment has the added effect of removing bicarbonate as carbon dioxide during the acid cycle. As discussed in Volume 1, bicarbonate concentration is an important factor in determining MFT behavior (through the control of pH), and at pH's above or below 8.4 (the bicarbonate buffer pH), the bulk properties of MFT are improved in terms of dewatering behavior, and the clay flocs or aggregates are less dispersed (stronger). Calcium addition via gypsum (no pH swing) also changes the aggregate structure [12], producing a stronger material that can dewater relatively rapidly with an applied stress. Figure 2-3 shows the segregation boundary of MFT and sand mixtures as a function of sand, water and fines content. The strength of the MFT floc structure determines the amount of sand that can be supported by the fines without segregation. Figure 2-4 shows how this boundary changes with chemical treatment. The effectiveness of freeze thaw processes is also a function of the water chemistry.

Even though the Clark MFT structure is altered by chemical means (either pH adjustment or calcium addition, or both), consolidation is still very slow. With the increase in strength of the floc structure, however, the consolidation can be enhanced by the application of a force or stress on the flocs. This can be achieved by an internal surcharge (the creation of non segregating tailings), or by an external surcharge (sand layering), or *via* freeze-thaw strains. Laboratory and field tests have demonstrated the effectiveness of NST in providing an internal surcharge to consolidate MFT [16–18].

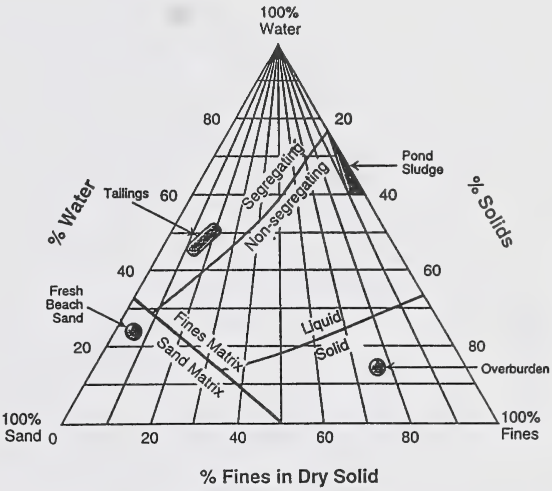

FIGURE 2-3. Triangle diagram relating fines, water and total solids content of oil sands tailings suspensions and the resulting segregation boundary. This segregation boundary is a function of the sand to fines content, as well as the suspension water chemistry, and is an indication of the strength of the fines matrix (the floc or aggregate structure).

Freeze thaw processes can also be thought of as a mechanical effect. During freezing, ice crystals form which force clay particles together and disrupt the floc structure. The formation of ice crystals removes water from the MFT suspension which increases the ionic strength of the remaining pore water, leading to chemical disruption of the aggregate structure as well. The mechanical effect is thought to be more of a factor than the increase in ionic strength [15]. The efficiency of the freeze thaw process will depend upon the relationship between the size of the ice crystals that are formed and the size of the flocs or aggregates that are being squeezed into peds. Laboratory and field testing has shown that winter temperatures in Fort McMurray will result in efficient water release with freeze-thaw [19–21]. Figure 2-4 shows the enhanced freeze thaw water release with changes in MFT water chemistry (OSLO *versus* Clark).

2.4 Summary

Clark MFT is a weakly flocculated suspension of clays that is at least partially electrostatically stabilized [22]. The properties of this clay suspension are determined by the behavior of the hydrophilic clays and are controlled by the water chemistry. Ionic concentration and pH are important factors in determining the floc structure, as well as bicarbonate ion concentration. Bicarbonate is the most abundant anion, and determines the pH (and therefore the bulk proper-

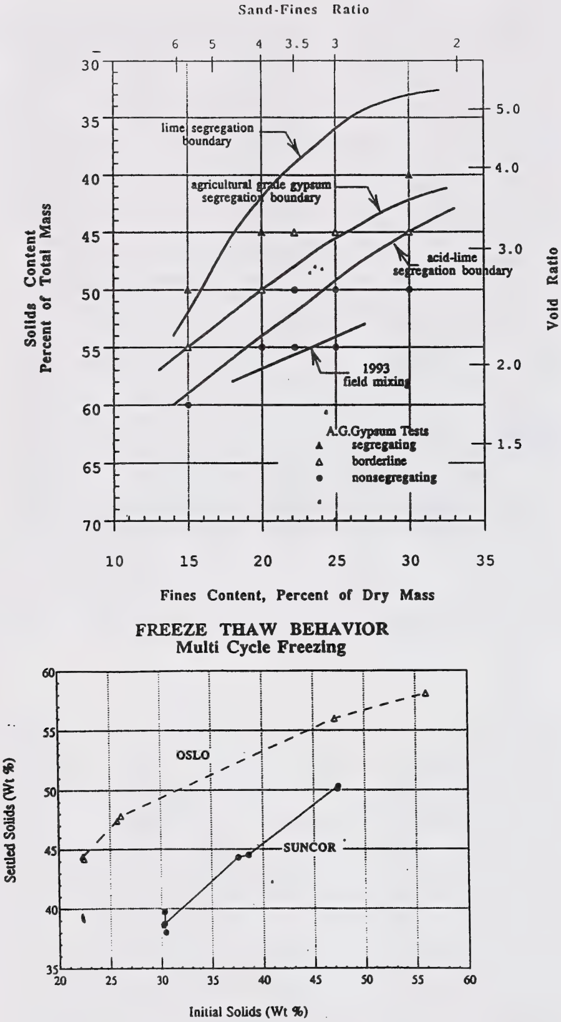

FIGURE 2-4. Changes in the segregation boundary with various chemical treatments. By changing the water chemistry, the floc structure and therefore the strength of the fines matrix is changed, resulting in changes in the segregation boundary. These floc structure changes are also reflected in the freeze thaw behavior of the tailings suspensions with varying water chemistry (OSLO *versus* Clark MFT shown).

ties) of the MFT. The Clark MFT floc structure has been found to be too weak to support a surcharge which could cause consolidation by gravity. This floc or aggregate structure results in a fluid material with very low permeability where pore pressures will not dissipate in any reasonable time frame.

A chemical or physical initiation can change the floc structure of the Clark MFT to a suspension that can be consolidated. Without any chemical manipulation of the floc structure, dewatering or volume reduction methods are unlikely to be successful. Once this stable structure is changed, however, a variety of methods can be used to enhance consolidation and dewatering. Water chemistry manipulation coupled with mechanical stresses can prevent formation of, or change, the Clark MFT structure and produce a suspension that, although it has similar solids content, is able to support a surcharge or will otherwise eventually consolidate. With calcium addition it is possible to produce a floc or aggregate structure strong enough to support a surcharge and thereby enhance dewatering by the creation of nonsegregating tailings or with sand layering. Freeze thaw strain also physically changes the floc structure to release water and can be effective both with chemically treated or untreated MFT. Many of these chemical or physical treatments have been successfully demonstrated on a large scale [18,22].

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3.0 VOLUME REDUCTION OF CHWE FINE TAILINGS UTILIZING NONSEGREGATING TAILINGS

3.1 Summary

Due to its gap-graded nature, the majority of the fine particles in the oil sand tailings stream segregate from the main sand deposit and form a fluid deposit called "fine tailings." The accumulation of large quantities of fine tailings leads to increased operating costs and a long term reclamation challenge. To prevent fines segregation, it is necessary to modify the tailings stream by increasing the solids content, increasing the fines content, or changing the apparent grain size of the fines. Flocculating or aggregating the fines with chemical agents, such as lime, sulphuric acid, $CaSO_4$, flyash (mainly $CaSO_4$) and their combinations, can change the tailings stream into a nonsegregating slurry. It is possible to produce nonsegregating tailings deposits with a wide range of engineering properties. Particular properties can be selected by adjusting the weight ratio of sand to fine mineral particles (called the sand to fines ratio, "SFR"). The selection of a SFR for a deposit is the optimization of a desire to incorporate a specified quantity of fines in the deposit while achieving consolidation and strength performance criteria within desired time frames.

The following summary documents the major conclusions based on extensive laboratory and field pilot testing of nonsegregating (NST) mixtures. For more detailed discussion, the reader is directed to the relevant sections which follow the summary.

3.1.1 Production of Nonsegregating Tailings

The effectiveness of chemical treatment in producing NST depends upon the solids and fines contents of the tailings stream and the gradation. Due to the low solids and fines of the Suncor plant tailings, it appears necessary to thicken the tailings stream for NST purposes. Syncrude plant tailings stream may be used directly for producing NST using either lime or $CaSO_4$.

In field operation, chemicals may be added to and effectively mixed in the tailings line using orifice or other in-line mixers. NST mixtures appear to be durable and, thus, are not susceptible to breakdown upon vigorous and prolonged mixing. Longer mixing time appeared to yield NST with better properties in sedimentation and initial consolidation. The temperature of the tailings stream does not have significant influence on the segregation or consolidation behavior of NST. The head loss of water through both mixers is about 80% of the head loss for raw or limed tailings. The head loss in the downstream pipe appears to be about the same for water, raw tailings and limed tailings.

3.1.2 Deposition of NST

A low energy discharge environment is important in producing nonsegregating tailings deposits under dynamic depositional conditions. Air entrainment during deposition causes some segregation of fines from the main deposit. The vertical, submerged discharge method is effective in dispersing energy and, as a result, preventing air entrainment. Discharge of NST in sub-aerial, sub-water or sub-MFT seems to have little effect on the behavior of the NST deposit.

When deposited at solids contents greater than 55%, NST flowed like a uniform viscous slurry.

It formed a very flat deposit with an average slope of 0.1 to 1%. The slopes of the NST deposits tended to decrease as the discharge rate (flow velocity) of the tailings increased. The flat slope of the NST deposit was sufficient to allow the release water to flow on the surface to the toe of the deposit.

The runoff water released from NST deposits was generally mineral free.

3.1.3 Consolidation/Volume Reduction of NST Deposits

With the addition of chemicals, fine particles in oil sands tailings become flocculated or coagulated. This process enhances the permeability of the tailings especially at relatively low solids contents (high void ratios). As a result, nonsegregating tailings undergo a significant volume reduction during sedimentation and initial consolidation which commences almost immediately after deposition with clear decant water released on top of the deposit. The rate of initial consolidation largely depends on the fines content of the tailings. Once the fines content is greater than 25% (sand to fines ratio smaller than 3), the rate of consolidation decreases considerably. It also depends on the type and concentration of additives and the length of mixing time. There is usually an optimum additive concentration at which the tailings yield the best rate of initial consolidation. Longer mixing time tends to produce tailings with better initial consolidation behavior.

The amount of solids content increase or volume reduction of NST during initial consolidation depends upon the initial solids and fines contents. Tailings with high initial solids yield high effective stresses and, consequently, consolidate to higher solids than low initial solids tailings. The field and laboratory tests indicate that approximately two-thirds of the ultimate volume change or settlement takes place during sedimentation and initial consolidation. The vertical effective stresses at this stage of volume reduction are about 1 to 2 kPa.

The amount of volume reduction in NST decreases as the tailings consolidate under increased effective stresses especially when a stable grain structure is reached. Similarly, the permeability of NST decreases as the void ratio of the tailings decreases due to consolidation.

When a tailings layer is placed, the soil structure of the layer immediately below collapses and the effective stress drops to zero. Channeling or quicksand action (rapid upward flow of water keeping soil at zero effective stress) may take place in the top layer immediately after placing. This would result in rapid pore pressure dissipation not only in the top layer but also in the layer below. Allowing the tailings to fully (or mainly) consolidate before the next deposition appeared to be beneficial in terms of the consolidation behavior of the tailings deposit.

Under continuous deposition (10 cm per day, for example), the average solids content and the degree of pore pressure dissipation remained nearly unchanged after the first five layers. The effective stress at the bottom increased slowly. As soon as the deposition stopped, the solids content and the pore pressure dissipation increased steadily.

3.1.4 Stress-Strain Behavior and Strength

In geotechnical terms, the NST mixtures are typically non-plastic. However, some of the higher fines content NST mixtures have liquid limits in the range of 12% to 15% while plastic limits range between 10% to 11%.

Laboratory prepared nonsegregating tailings samples, which are normally consolidated under isotropic or k_0 conditions to a maximum effective stress of 200 kPa, showed strain softening behavior, that is, deviator stresses decreased with increases in axial strains after peak. The softening behavior was more obvious in lower fines tailings. Pore pressures developed during undrained compression, indicating a contractive behavior of the NST samples at the stress range used in the tests.

Tailings samples consolidated under k_0 conditions, in which the tailings are consolidated in the vertical direction only with no horizontal deformation, showed a stress-strain behavior similar to the isotropically consolidated (CI) tailings during undrained compression. Due to the deviator stresses developed during k_0 consolidation, the tailings reached the peak strength at axial strains considerably smaller than the CI tailings.

Preliminary results indicated that the void ratio-effective stress relationships of the NST consolidated to a maximum 80 kPa effective stress lie to the right of the steady state line defined in undrained triaxial tests. It means that, when normally consolidated, the tailings are potentially liquefiable, rapidly developing excess pore pressures and losing strength if subject to large strains or cyclic loading conditions. The liquefaction potential of NST, however, may be reduced by means of over-consolidation (overburden surcharge) or aging. More detailed and comprehensive studies are required to further evaluate the liquefaction potential of NST and its controlling factors.

The undrained shear strengths of NST, peak and remoulded strengths measured using vane shear testing, increase as the fines content and effective consolidation stress increase. The sensitivity (a ratio between peak and remoulded shear strength) of the tailings seems to have no clear trend of variation. The undrained shear strengths measured in field trials are in general agreement with laboratory measurements. The profiles of the undrained shear strength obtained in the field show an increase in peak and remoulded strength with depth in the tailings, indicating that the tailings are consolidating and thus gaining strength. The sensitivity of the nonsegregating tailings is generally in the range of 2 to 4, which is also similar to that found in the laboratory on similar tailings mixes.

Although the strength database is in its early stages, several observations can be made.

- The NST deposit will have a significantly increased strength profile with depth compared to MFT.
- The strength of NST deposits near the surface will likely be sufficient to sustain the small surcharge loading necessary to prepare it for larger construction traffic which could be required for a fully reclaimed surface.
- Any desiccation through drying or freeze/thaw action will further increase the possibility of supporting construction traffic.

Therefore, indications at this stage, with respect to the use of NST technology in creating a reclaimable dry landscape, are encouraging.

3.2 Background

Due to its gap-graded nature, the majority of the fine particles in the oil sands tailings stream segregates from the main sand deposit. To prevent fines segregation, it is necessary to modify the tailings stream by increasing the solids content, increasing the fines content, or changing the grain size of the fines. Flocculating or aggregating the fines with chemical agents, such as lime, sulphuric acid, $CaSO_4$, flyash (mainly $CaSO_4$) and their combinations, can change the tailings stream into a nonsegregating slurry.

A nonsegregating tailings stream is defined as a tailings mixture in which the fine and sand particles settle simultaneously to form a uniform deposit. Nonsegregating tailings can be produced in different ways. The technique discussed in this chapter is to flocculate or coagulate the fines by adding chemicals to the tailings stream. The nonsegregating mixtures have much improved sedimentation and consolidation characteristics over MFT, resulting in a significant volume reduction shortly after deposition. Once a stable soil structure is reached as a result of the fast initial volume reduction, the deposit will consolidate under self-weight or applied loads similar to a natural deposit.

There are several tailings management scenarios in which nonsegregating tailings technology may be implemented. The three main tailings streams suitable for nonsegregating tailings production are:

1. the total plant tailings stream
2. the combination of primary tailings (without the middlings stream) and MFT
3. the combination of MFT and the underflow of hydraulically cycloned plant tailings

Considering the vast volume of the oil sands tailings, cost of the chemicals used for producing NST must be reasonably low in order to make the technology economically feasible. The possibilities of using lime, sulphuric acid, $CaSO_4$ and different types of flyash (mainly $CaSO_4$) and their combinations have been investigated. $CaSO_4$ is a major component of the waste product from scrubbers which remove SO_2 from power plant stacks. The use of different chemicals not only affects the geotechnical behavior of NST, but also affects the chemistry of the release water.

The issues that had to be resolved by the NST research included:

- What is required to produce nonsegregating tailings and what are the optimum additive concentrations?
- How do we effectively and efficiently mix chemical additives with tailings in a field operation?
- How does NST behave during deposition and what is the slope of the deposit?
- How fast does NST settle and consolidate and what are the volume changes?
- How do operational options, such as multiple layer or sub-MFT deposition, affect the behavior of NST?
- What are the stress-strain characteristics and strengths of consolidated NST?
- What is the quality of the water released from NST deposits?

The above issues will be discussed in the following sections except for the release water quality which is discussed in Chapters 5.0 and 6.0 of this volume.

3.3 Production of Nonsegregating Tailings

Segregation depends upon the solids content of the tailings stream, the gradation of the solids and the energy of the depositional environment. Figure 3-1 shows the segregation boundary for an untreated raw oil sands tailings stream. Above this boundary the solids content is sufficiently low that fines segregation can occur while below the boundary, no appreciable segregation takes place.

Figure 3-1 also shows the average solids content of the tailings streams from both Syncrude and Suncor operations. The solids content of Syncrude plant tailings varies from 35% to 62% with an average of 56%; the solids content of Suncor plant tailings varies from 30% to 60% with an average less than 40%. It is apparent that the entire range of both Syncrude and Suncor oil sands tailings lies above the segregation boundary. The further the range is above the segregation boundary, the greater will be the amount of fines segregation.

To prevent fines segregation, it is necessary to modify the tailings stream by increasing the solids content and/or by increasing the fines content. A change in grain size distribution can also be accomplished by changing the apparent particle size of the fines. Flocculating or aggregating the fines with a chemical agent can then change the tailings stream into a nonsegregating slurry.

Scanning electron microscopy and sedimentation tests have been conducted on lime treated Syncrude plant tailings and the results indicated that the lime forms aggregates of the fines in the 10 to 100 micron particle size range.

The efficiency of various chemicals in preventing segregation varies as they are used for different tailings materials. The following combinations of tailings and chemicals have been proven successful in preventing segregation:

- quick lime with Syncrude plant tailings
- quick lime with Syncrude plant tailings spiked with middling fines (from line #73)
- quick lime with Syncrude plant tailings spiked with mature fine tails
- sulphuric acid with Syncrude plant tailings
- $CaSO_4$ on Syncrude plant tailings + MFT
- combination of acid and lime with Syncrude plant tailings
- combination of acid and $CaSO_4$ with Syncrude plant tailings
- combination of acid and lime with Suncor plant tailings with relatively high solids and fines

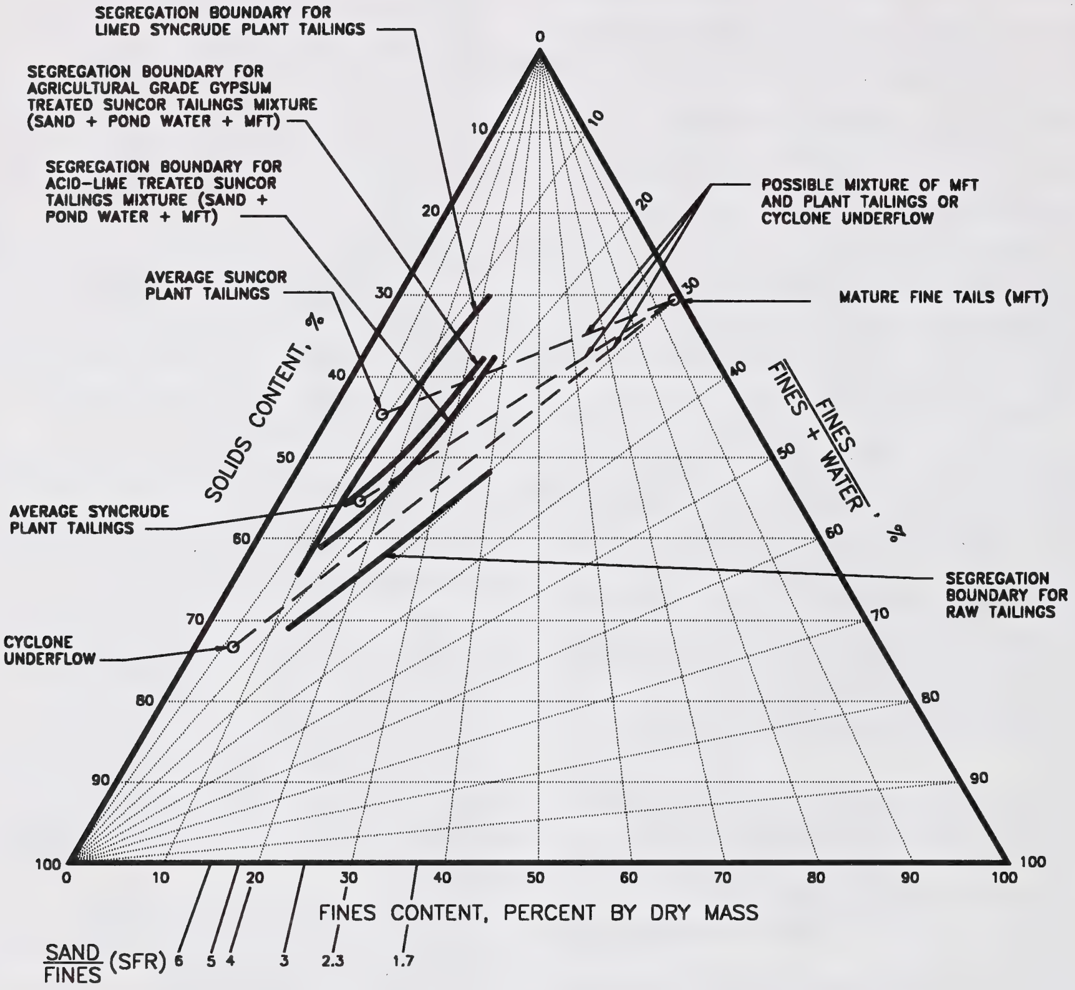

FIGURE 3-1. Slurry properties diagram.

- combination of acid and flyash ($CaSO_4$) with Suncor plant tailings with relatively high solids and fines
- combination of acid and lime with Suncor tailings sand + MFT + pond water
- combination of acid and flyash ($CaSO_4$) with Suncor tailings sand + MFT + pond water

It was found in laboratory segregation tests that it is more difficult to prevent segregation as the solids and fines contents decreased in the tailings stream. The amounts of chemical additives required to treat the tailings are determined such that not only a nonsegregating tailings stream is produced, but reasonable sedimentation/consolidation characteristics and acceptable release water quality are obtained. Typical segregation boundaries for chemically treated oil sands tailings are presented in Figure 3-1.

It is seen from the figure that, as the gradation of the tailings changes due to flocculation or coagulation, the segregation boundaries of the tailings move upwards. Consequently, nonsegregating tailings streams are produced at the range of solids content where the tailings are still pumpable. It is also seen from the figure that the average Suncor plant tailings are above the segregation boundary for acid-lime treatment. Therefore, in order to produce nonsegregating tailings, the plant tailings need to be thickened for NST disposal purposes.

The effects of mixing time and temperature on the behavior of NST have been studied for the case of quick lime or sulphuric acid treatment of Syncrude plant tailings. Parallel tests were conducted using different temperatures (20°C *versus* 60°C) and different mixing times (up to 30 minutes). The effect of mixing time on the viscosity of the mixes was also investigated using a Marsh funnel viscometer. The following observations have been made:

- Increases in mixing time and temperature did not increase segregation of the mixes.
- The mixing temperature did not have significant influence on either sedimentation or self-weight consolidation of NST.
- The rate of sedimentation and initial self-weight consolidation increased as the mixing time increased from 5 to 30 minutes. However, the mixes with shorter mixing time appeared to consolidate faster in the later self-weight consolidation stage.
- The funnel viscosity of the mixes decreased as the mixing time increased for the lime treated tailings. The viscosity of the acid treated tailings remained nearly unchanged when the mixing time was greater than 5 minutes.

To study the technology of mixing chemical additives to the tailings within a pipeline, a series of tests was performed on lime treated Syncrude tailings using an orifice and an Aqua-Shear (a commercial brand name) mixer. A laboratory scale "tailings line" of 3.8 cm (1.5 inch) was assembled and a variable speed progressive cavity pump was used to pump the tailings. Lime milk was injected into the tailings in the pipeline at a constant rate. The minimum residence time for the lime was 2.5 seconds in the pipeline before the tailings were deposited into a flume and a maximum residence time of 10 minutes was used in separate tests to examine the durability of the NST. The following observations and conclusions were obtained from the in-line mixing tests:

- Orifice and Aqua-Shear mixing achieve a uniformly mixed NST. No obvious difference in NST behavior was seen between the two mixing methods.
- Longer mixing time appeared to yield NST with better properties. One pass through the mixer, with 2.5 seconds residence time in the downstream pipe before deposition was adequate to prevent segregation but did not achieve a mixture with optimal sedimentation and consolidation characteristics.
- This finding also indicates that the flocculated or coagulated fines are not susceptible to breakdown upon vigorous and prolonged mixing. In other words, the NST mixtures are durable.
- The head loss of water through both mixers is about 80% of the head loss for raw or limed tailings. The head loss in the downstream pipe appears to be about the same for water, raw tailings and limed tailings.

Nonsegregating tailings mixes were also obtained in a field scale trial conducted at the Suncor Fort McMurray site during the summer of 1993, using a small pilot plant. A hydrocyclone was connected to a slipstream of one of the main tailings lines. The underflow from the hydrocyclone discharged into a tank where it was mixed with mature fine tails from a nearby tailings pond. The mixture was then pumped to the test pits through a pipeline into which acid and lime were injected and mixed with the tailings stream as it travelled within the pipeline.

The acid and lime treated tailings were deposited into 25 m × 150 m by 5 m deep test cells and samples taken at different locations and depths were analyzed. There was no evidence of segregation, as the fines content of each layer remained relatively constant along the length of the cell and similar to the fines content of the tailings mixture. The relatively small difference in fines content between layers indicates that the pilot plant was successfully controlled to produce NST within a specified composition range.

3.4 Deposition of NST

A series of dynamic laboratory flume tests was conducted on chemically treated Syncrude and Suncor tailings to develop discharge techniques for NST and to study flow characteristics of NST under different depositional energy environments. The option of depositing NST under water was also investigated in the flume tests.

Air entrainment during deposition caused some segregation of fines from the main deposit. The vertical, submerged discharge method was the most effective in dispersing energy and, as a result, preventing air entrainment. Horizontal discharge was not effective as it caused hydraulic jumps, resulting in high turbulence and some segregation. The treated tailings partially segregated when the tailings were turbulently mixed with water in underwater deposition.

When deposited at solids contents greater than 55%, NST flowed like a uniform viscous slurry. It formed a very flat deposit with an average slope of 0.1 to 1%, considerably different from untreated tailings which formed a sand beach with 5 to 10% slopes and a flat fine tails deposit. The flat slope of the NST deposit was sufficient to allow the release water, due to sedimentation of the tailings, to flow on the surface to the toe of the deposit. The solids content of the runoff water released from NST was less than 1% and the solids (fines) remain flocculated as they settled out rapidly.

Deposition of NST was also studied in the field scale trial at the Suncor site during the summer of 1993. The tailings stream was discharged through a slotted pipe along the bottom of one end of the test pits. The discharge point soon became submerged. It was observed that the tailings formed a slope of about 0.1%.

Subaqueous and sub-MFT discharge methods were also investigated in the field trial program. It was found that the method of discharge (sub-aerial, sub-water or sub-MFT deposition) had little effect on the behavior of the NST deposit. Some fines were discharged into the water in the subaqueous deposition method (enough to make the water cloudy), but were flocculated and quickly settled out to leave clear water in less than 1 day. The mature fine tails above the NST appeared to have little effect on the rate of sedimentation/consolidation of the NST. It is unclear what effect the MFT may have on the chemistry of the underlying NST.

3.5 Consolidation/Volume Reduction of NST

After a chemically treated nonsegregating tailings stream is deposited in slurry form, it goes through two main stages of dewatering. First, the tailings particles settle at its low initial solids content (high void ratio) to form a structured deposit with grain to grain contact. This phase is identified as sedimentation/initial self-weight consolidation or simply as initial consolidation. A large volume of water is released from the tailings as a result of this phase of volume reduction. Then, after a grain structure is formed, the tailings deposit continues its long process of volume reduction under self-weight or applied loads. Pore water within the grain structure is pushed out under excess pore pressures and the solids content of the tailings deposit continues to increase (void ratio decrease) consequently. Since the self-weight of the tailings increases during the course of tailings deposition, the process of self-weight consolidation continues, extending after deposition of NST is complete.

Laboratory test programs, including standpipe and consolidation tests, have been conducted to study the sedimentation and consolidation characteristics of Syncrude and Suncor nonsegregating tailings and to investigate the influence of fines content on the consolidation behavior. Special tests were also performed to investigate the volume reduction behavior of NST under multiple layer deposition, in which a layer of NST was allowed to fully consolidate before a next layer is deposited, and sub-MFT deposition, in which NST was deposited and allowed to consolidate under a layer of MFT.

Hundreds of 90 cm high standpipe tests were performed on Syncrude and Suncor tailings treated with different types of chemicals. Sedimentation of NST started almost immediately after deposition with clear decant water released on top of the deposit. The tailings-water interface usually dropped significantly during the first 24 to 48 hours after deposition and then slowed down thereafter as self-weight consolidation began. Pore pressures in the tailings were measured in some tests.

Nonsegregating tailings deposits in the 1993 Suncor field trial had sedimentation and initial consolidation characteristics similar to that in laboratory standpipe tests.

A series of step load large strain consolidation tests has been conducted to evaluate the long term consolidation behavior of NST under self-weight and applied loads. The tests were conducted on quick lime treated Syncrude tailings, and acid/lime and acid/flyash treated Suncor tailings mixtures. A range of fines contents from 12% (sand/fines ratio SFR = 7) to 35% (SFR = 2) was tested. Due to the fact that NST is made up of a large proportion of sand particles, large diameter (17 cm or larger) consolidation cells were used to minimize the arching effect which could otherwise be substantial at high stresses.

The constant head permeability tests were conducted between load steps to measure permeability of the consolidated NST. Vane shear tests were also performed in some consolidation tests to measure the undrained shear strength of the tailings.

From standpipe and large strain consolidation tests, sedimentation and consolidation characteristics of NST have been determined. They include two aspects, compressibility and permeability (hydraulic conductivity), and their changes with effective stress during consolidation of NST. Compressibility of NST is usually represented in void ratio (e) *versus* effective stress (p') plots, with the slope of e–p' plots becoming smaller as NST consolidates, reflecting densification of the tailings structure. The change in permeability of NST is often presented in permeability *versus* void ratio plots, which also reflect changes in NST structure.

Figures 3-2 and 3-3 summarize the compressibility and permeability of quicklime treated average Syncrude tailings and acid/lime treated Suncor tailings mixtures at sand/fines ratios of 3 and 4. The consolidation behavior of other NST is contained in technical reports to Syncrude and Suncor.

From laboratory standpipe and large strain consolidation tests and results of a field trial, major findings on sedimentation and consolidation characteristics of NST are summarized below:

- With the addition of chemicals, fine particles in oil sands tailings are flocculated or coagulated. This process enhances the permeability of the tailings especially at relatively high void ratios. As a result, nonsegregating tailings undergo a significant volume reduction during sedimentation and initial consolidation which commences almost immediately after deposition and is complete within a short period of several days to a few weeks. Field and laboratory tests indicate that approximately two-thirds of the ultimate volume change or settlement takes place during sedimentation and initial consolidation. The vertical effective stresses at this stage of volume reduction are about 1 to 2 kPa.

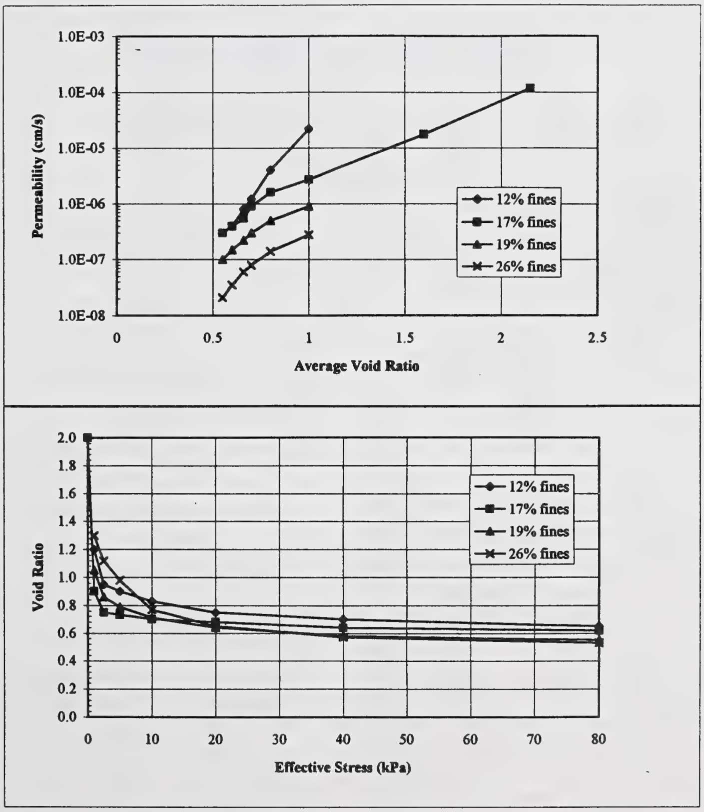

FIGURE 3-2. Consolidation characteristics of lime treated Syncrude plant tailings.

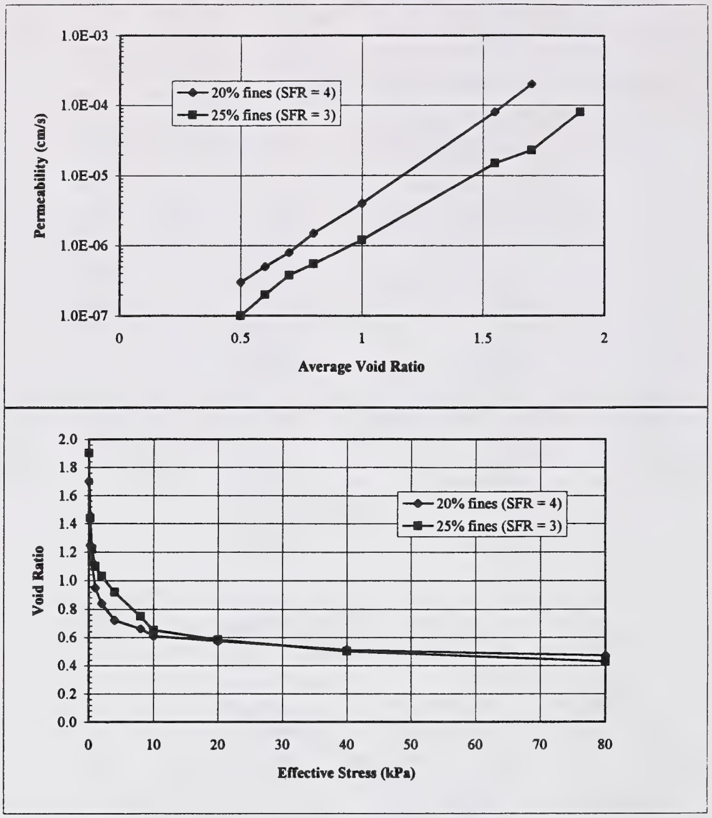

FIGURE 3-3. Consolidation characteristics of acid-lime treated Suncor plant tailings.

- The rate and amount of sedimentation and initial consolidation depend on the initial solids content (void ratio), the amount and type of fines (*i.e.*, clay or silt), the type and amount of chemicals, and the mixing procedure and mixing time. Laboratory tests show a large difference in NST sedimentation and initial consolidation rates depending on the fines, the chemicals and the mixing procedures used. It was found that, once the fines content is greater than 25% (sand to fines ratio smaller than 3), the rate of consolidation decreases considerably. It was also found that sufficient mixing time tended to yield NST with better sedimentation and initial consolidation characteristics.
- The amount of volume reduction decreases as the tailings consolidate under increased effective stresses especially when a stable grain structure is reached. In other words, the compressibility of the tailings decreases as the effective stress increases.
- Permeability of NST varies with the fines content and the void ratio. As indicated in Figures 3-2 and 3-3, the permeability decreases as the void ratio decreases and as the fines content increases. For the same sample, the permeability decreased as the void ratio of the tailings decreased due to consolidation.
- For most of the consolidation tests, the void ratio of the nonsegregating tailings decreased to less than 0.9 after consolidation under 2.5 kPa applied load.
- As shown in all multilayer standpipe tests, when a tailings layer was placed, the soil structure of the layer immediately below collapsed and the effective stress dropped to zero.

- In multi-layer standpipe tests, the lower layers consolidated faster than the upper layers possibly due to the fact that the lower layers had a much smaller compressibility than the upper layers.
- It appears that two 45 cm (1.5 foot) layers consolidated more rapidly than a single 90 cm layer for SFR = 4 and higher. Allowing the tailings to fully (or mainly) consolidate before the next deposition appeared to be beneficial in terms of consolidation behavior of the tailings deposit.
- Under continuous deposition (10 cm per day), the average solids content and the degree of pore pressure dissipation remained nearly unchanged after the first five layers. The effective stress at the bottom increased slowly. As soon as the deposition stopped, the solids content and the pore pressure dissipation increased steadily.
- In sub-MFT standpipe tests, clear decant water released from the NST appeared on top of the MFT surface within 24 hours. The MFT cap did not seem to prevent the release water from flowing through. The results of sub-MFT standpipes were compared with parallel standpipe tests of NST deposited subaerially. It was found that both the rate and amount of volume changes and the final solids content profiles (after full consolidation) in NST deposited under MFT are similar to the NST deposited subaerially.

Numerical modelling on the rate and amount of consolidation of NST deposits was conducted using an integrated finite strain consolidation program. The program used the relationships of void ratio *versus* effective stress and void ratio *versus* hydraulic conductivity, which were determined from laboratory standpipe and consolidation tests. The program was used to model and compare with the consolidation behavior of NST in the tanks and pits filled during the 1993 field program at the Suncor site. The program would then be used to predict the performance of NST in a commercial operation.

The solids content profiles from the numerical modelling were somewhat higher than the actual profiles for all of the cells and tanks, especially at the top and bottom of the deposits (Figure 3-4). The overall conclusion is that predictions performed with laboratory derived consolidation parameters tend to be somewhat more optimistic than the field performance. This conclusion is, however, preliminary due to inaccuracies in the field data, especially the pore pressure measurements. The differences between predicted and measured solids contents in the cells and tanks became smaller as the NST deposits consolidated with time.

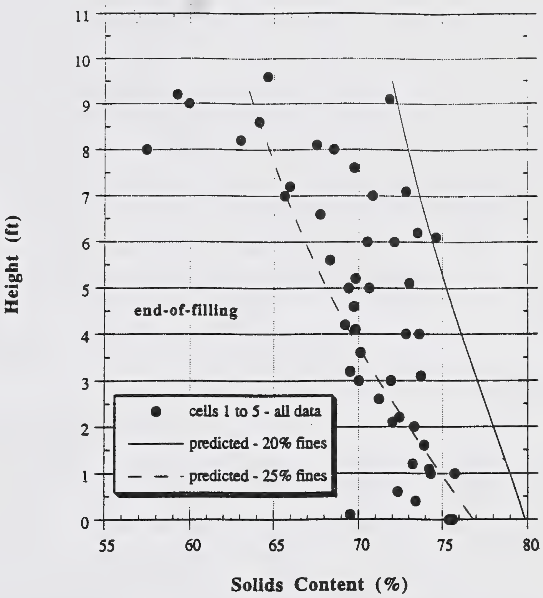

FIGURE 3-4. Predicted and actual solids content profiles at the end of filling, cells 1 to 5, Suncor NST.

3.6 Strength Behavior of NST

Stress-strain behavior and shear strength of consolidated nonsegregating tailings have been studied through undrained triaxial compression tests on laboratory prepared NST samples and vane shear tests on both laboratory and field consolidated NST deposits.

Undrained triaxial compression tests were performed on laboratory prepared Suncor nonsegregating tailings at 20% and 25% fines contents (SFR 4 and 3, respectively) with acid-lime treatment. The main objectives of the triaxial tests were to study the stress-strain behavior and pore pressure response of a consolidated NST deposit at different effective stresses and to evaluate its liquefaction potential, which is associated with rapid increases in pore pressures, dramatic loss in strength, large deformations and possibly flow failures in saturated sand deposits (especially loose sand deposits). An additional objective of the tests was to obtain shear strength parameters of NST.

A series of triaxial tests has been conducted on NST samples under effective confining stresses up to 200 kPa. The sample was consolidated either under a predetermined isotropic confining stress or under a k_0 condition (one dimensional consolidation without lateral deformation) and sheared without drainage. The following findings were obtained from the triaxial tests on laboratory prepared Suncor NST samples (SFR 3 and 4):

- All tailings samples isotropically (CI) or k_0 (CK) consolidated to effective stresses up to 200 kPa, showed strain softening behavior, that is, deviator stresses decreased with increases in axial strains after peak. The softening behavior was more obvious in lower fines tailings (SFR 4 vs. 3). At large confining stresses, the deviator stresses tended to increase slightly as approaching the steady state.
- Pore pressures developed during undrained compression in all tailings samples, indicating a contractive behavior of the NST samples at the stress range used in the tests. The axial strains required to develop the maximum pore pressures increased with increases in effective confining stress.
- Tailings samples consolidated under k_0 conditions showed a stress-strain behavior similar to the isotropically consolidated tailings during undrained compression. Due to the deviator stresses developed during k_0 consolidation, the tailings reached the peak strength at axial strains considerably smaller than the CI tailings. Similarly, the CK tailings developed the maximum pore pressures at smaller axial strains than the CI tailings during undrained compression.
- The k_0 value of the tailings was estimated from the k_0 consolidation test to be about 0.8 at low effective stress levels (σ_1'<60 kPa) and 0.45 at high stress levels (σ_1'>60 kPa).
- Strength envelopes from the triaxial tests on the CI tailings gave the peak and large strain effective friction angles of 23° and 35° for the tailings of SFR 4 and the friction angles of 21° and 37° for the tailings of SFR 3. The effective friction angles of the CK tailings samples were 23° at the peak and 39° at the steady state, similar to the CI samples (SFR = 4).
- Upon isotropic consolidation, the void ratio-mean effective stress relationships of the tailings sample with a same SFR fell on a straight line in the e-log p' space. At the same effective consolidation stress, the SFR 4 tailings consolidated to lower void ratios than the SFR 3 tailings. All tailings, however, finished to nearly the same e-log p' relationships at large strains.
- At the same mean effective stresses, the tailings under k_0 conditions consolidated to higher void ratios than the CI tailings.

In addition to the triaxial compression tests, shear strengths of Suncor NST were also measured using vane shear tests on laboratory consolidated and field NST deposits. The findings from these vane shear tests are summarized in the following:

- For each tailings specimen, the results from three independent tests were consistent. The stress-strain relationships and the undrained shear strengths (peak and remoulded) from the three tests were similar to each other, indicating the results are repeatable.
- Both the peak and the remoulded strengths of the tailings increased as the effective consolidation stress increased.
- The undrained shear strength (peak and remoulded) of the tailings increased as the fines content increased for the same normal effective stress.
- Generally speaking, the ratio between the peak undrained shear strength and the effective consolidation stress (c_u/p') decreased as the effective consolidation stress increased. However, for the acid and lime treated tailings, the c_u/p' ratio remained the same when the fines content was greater than 20%.
- The c_u/p' ratio for the remoulded strength showed the same trend of decreasing as the effective consolidation stress increased even though the magnitude was smaller.
- The sensitivity (a ratio between peak and remoulded shear strength) of the tailings did not show a clear trend of variation. The tailings at 20% of fines appeared to have higher sensitivity than other mixes.
- During the 1993 Summer field trial at the Suncor site, undrained shear strengths ranging from 1.5 to 2.5 kPa were measured close to the base of the test tanks where the total NST thickness varies from 3.7 to 4.3 m (12 to 14 ft); strengths from 0.6 to 1.0 kPa were obtained close to the base of the test pits where the NST thickness ranges from 1.5 to 3.0 m (5 to 10 ft). The undrained shear strength profiles generally show an increase in peak and remoulded strength with depth in the tailings. This indicates that the tailings are consolidating and thus gaining strength. The sensitivity of the nonsegregating tailings is generally in the range of 2 to 4, which is similar to that found in the laboratory on similar tailings mixes.

3.7 Appendix I. Research Related to Nonsegregating Tailings

The following is a review of the literature that is readily accessible on the use of chemicals to treat total tailings (the complete tailings stream). There may be research or proposed methods of treatment that have not been included here. Nevertheless, the following provides a representation of the work that was performed prior to the current nonsegregating tailings studies.

3.7.1 Early Investigative Studies on Chemical Treatment of Total Tailings

A patent by Liu *et al.* (1980) suggests a method of treating total tailings before vacuum filtration. Quick lime was found to work the best.

Yong *et al.* (1981) Laboratory and Pilot Testing of Acid-Lime treatment of Sand Surcharged Fine Tailings. McGill University project at Suncor.

Erno and Hepler (1981) investigated the use of various additives, including lime and sulphuric acid, to enhance pressure filtration of total tailings.

C-H Synfuels (1984) studied the potential of treating tailings and then depositing the tailings using the thickened discharge method of Robinsky.

Marsh and Shaw (1984) performed some small scale flume tests on tailings treated with lime.

Scott and Chichak (1986) determined the minimum concentrations of lime or sulphuric acid to prevent segregation of oil sand tailings in standpipes and small scale flumes.

A review of the literature indicates that the early work was focused on obtaining tailings that are suitable for vacuum or pressure filtration or for building beaches and slopes. Filtration was not an economical method to treat oil sand tailings. To build beaches and slopes with tailings, the tailings must gain in strength rapidly. Tailings treated to make them nonsegregating form very flat slopes (less than 1%) due to the fines retained within the tailings structure.

Research since 1990 has been on determining the segregation boundaries, sedimentation and consolidation characteristics and chemical properties of the runoff of nonsegregating tailings.

3.7.2 Research on Nonsegregating Tailings (NST)

3.7.2.1 *Initial Laboratory Program on Limed Syncrude Tailings, 1990–1992*

A laboratory program was performed at the University of Alberta to determine the amount of lime required to prevent segregation of typical Syncrude tailings. Determination of the sedimentation and consolidation (self-weight and under applied loads) rates and hydraulic conductivity of tailings treated with the optimum lime concentration (Caughill, 1992). Initial predictions of the performance of NST when deposited in pit.

3.7.2.2 *In-Depth Laboratory Program on Limed Syncrude Tailings, 1992–1993*

An in-depth laboratory program was performed at the University of Alberta on limed Syncrude tailings. The first step in the program was to divide the Syncrude tailings stream into nine categories by solids and fines content. The segregation and sedimentation characteristics of the tailings were then determined according to category. The amount of segregation was determined by the amount of fines that were retained within the sand portion of the tailings, expressed as a percentage. Flume tests were performed to study: (1) the amount of segregation of NST after higher energy deposition; (2) methods of discharge to reduce turbulence, air entrainment and sudden energy drops; (3) beach slope of deposits as a function of discharge energy and solids/fines contents of the tailings; (4) behavior of NST during underwater deposition; (5) rate of water release (sedimentation) and solids content of release water; and (6) sedimentation of solids (fine solids) in release water.

Consolidation tests were performed to determine the compressibility and permeability of limed total tailings at different fines contents. The tests were performed to effective stresses of 80 kPa. Addition standpipe tests were also performed to determine the effects of effects of lime concentration, mixing energy, time and temperature. Chemical analyses of the release water were also conducted.

Additional flume tests were also performed to investigate in-line mixing of lime into the tailings stream and to determine the initial pumpability of NST. The tailings were piped through a hose within the laboratory and subsequently deposited into a flume. Two types of in-line mixers were tested, an orifice and an Aqua-Shear mixer. The efficiency of the mixing method was determined by measuring the properties of the deposit in the flume (segregation, consolidation, beach slopes, *etc.*) and the head loss across the mixer and across the length of the hose. The effects of over mixing were determined by pumping the treated tailings through a loop in the laboratory for up to one half an hour.

The results of the laboratory program are in 10 reports that are listed in the bibliography at the end of this report.

3.7.2.3 *Laboratory Test Program on Suncor Nonsegregating Tailings, 1993–1995*

An extensive laboratory test program was performed on Suncor nonsegregating tailings from 1993 to 1995. Three combinations of nonsegregating tailings were tested: (1) neat plant tailings; (2) a mixture of primary tailings and mature fine tailings (MFT); and (3) a mixture of cyclone underflow and MFT. These combinations represent most of the ways that nonsegregating tailings can be produced. Tailings with sand/fines ratios (SFR) from 6 to 2.3 were tested, with the majority of tests focused on SFR=4 and SFR=3 tailings.

A series of chemicals was tried as the testing evolved and new ideas came forward. The test program began by comparing the effectiveness of lime to treat tailings as well as sulphuric acid and lime to treat tailings. During the test program for acid and lime tailings, Suncor decided to explore the possibility of using the flyash from a fluidized bed boiler power plant that Suncor had planned to construct. The flyash product would be a calcium rich product that was both tested with and without the addition of sulphuric acid. After the completion of the flyash tests, Suncor had then decided to install a scrubber on the present power plant to reduce the emissions from the plant instead of constructing a new power plant. A by-product of the scrubber is a calcium sulphate rich product. The laboratory test program using calcium sulphate is currently underway at the University of Alberta as of March 1995.

The laboratory test program consisted of the following components:

- standpipe tests to study:
    1. segregation
    2. self-weight consolidation (solids content increases and pore pressure dissipation)
- flume tests to investigate:
    1. amount of segregation after higher energy deposition
    2. beach slope of deposits as a function of discharge energy and solids/fines contents of the tailings
    3. rate of water release and solids content of release water
    4. sedimentation of solids (fine solids) in release water
- large strain consolidation tests to study
    1. consolidation behavior of NST under applied loads (up to 80 kPa)
    2. permeability of NST at different void ratios
    3. estimates of undrained shear strength using the vane shear
- triaxial tests to analyze
    1. stress-strain behavior of consolidated NST under undrained conditions
    2. liquefaction potential of consolidated NST (isotropically or K_0 consolidated up to a maximum effective stress of 80 kPa)
- special considerations
    1. behavior of NST under multi-layer depositional conditions
    2. behavior of NST deposited beneath MFT

3.7.2.4 *Nonsegregating Tailings Field Trial, 1993–1994, Suncor Site, Suncor/Syncrude Tailings*

The field trial was performed on the Suncor site during the summer and fall of 1993. Six test pits and six large tanks were filled with Suncor tailings treated with sulphuric acid and lime. Two tanks were filled with Syncrude tailings treated with lime.

The primary objectives of the test program were:

1. geotechnical behavior (sedimentation, consolidation, *etc.*) of NST at field scale
2. characteristics of deposit (amount of segregation, flow rate, slope, *etc*)
3. investigate deposition procedures to produce nonsegregating tailings
4. material management and cyclone operation
5. procedures and techniques for mixing additives with tailings in a pipeline
6. pumpability of nonsegregating tailings
7. release water chemical analysis

The results of the field trail, in conjunction with the previous laboratory results, were used to predict the performance of NST when filling Pond 5 at Suncor. The predictions included: (1) rate of settlement of the surface of NST; (2) rate of consolidation/solids content profiles with time; and (3) rate of release water production.

The reports summarizing the field trial are listed in the bibliography.

3.7.2.5 *Nonsegregating Tailings Laboratory Testing Program, 1994–1995, Syncrude Tailings*

Three combinations of nonsegregating tailings were tested: (1) neat plant tailings; (2) a mixture of primary tailings and mature fine tailings (MFT); and (3) a mixture of cyclone underflow and MFT. Tailings with sand/fines ratios (SFR) from 5 to 3 were tested, with the majority of tests focused on SFR=4 and SFR=3 tailings.

Two chemical treatments were tested, acid and lime and $CaSO_4$. The work consists of standpipe tests and chemical analysis of the release water and was still in progress as of March 1995.

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Report 1	Characterization of Tailings and Additives
Report 2	One Metre High Standpipe Tests on Lime Treated Tailings
Report 3	One Metre High Standpipe Tests on Sulphuric Acid Treated Tailings
Report 4	Effects of Mixing Time and Temperature
Report 5	Flume Test Data
Report 6	Analysis of Flume Tests
Report 7	Two Metre Standpipe Tests
Report 8	Large Strain Consolidation Tests
Report 9	In-Line Mixing Tests on Limed Tailings
Report 10	Summary Report on Total Tailings Disposal Research Program

Laboratory Test Program on Suncor Nonsegregating Tailings, 1993–1995

Suncor Flyash Study Report

Nonsegregating Tailings Field Trial, 1993–1994, Suncor Site, Suncor/Syncrude Tailings

Suncor Nonsegregating Tailings — Summer 93 Field Experiment — Data Report on the Cell Filling and Monitoring Report

Suncor Nonsegregating Tailings — Summer 93 Field Experiment — Data Report on the Tank Filling and Monitoring Report

Suncor Nonsegregating Tailings — Summer 93 Field Experiment — Geotechnical Analysis Report for the Tank and Cell Filling Program

Suncor Nonsegregating Tailings — Summer 93 Field Experiment — Pond 5 Filling Report — Predictions for Filling Pond 5 with Nonsegregating Tailings

Summary Report on Initial Consolidation of Suncor Nonsegregating Tailings

4.0 FREEZE-THAW DEWATERING OF OIL SANDS FINE TAILINGS

4.1 Introduction

The use of ice crystal growth to separate water from impurities has been applied for decades to industrial processes. The phenomena acting within soil materials annually causes significant damage to foundation and transportation systems. Investigation of freeze-thaw as a means to dewater fine tailings was already underway by 1970. Although the process could be demonstrated to work, a feasible implementation strategy was not obvious. Nature only provides a single winter per year, and freezing of more than about 1 meter deep in a deposit per year could not be guaranteed. Therefore, the surface area requirements were unacceptably large to treat the volume of fine tailings accumulating each year.

There is currently a realization that many techniques may be required in concert to handle all of the fine tailings. Freeze-thaw is an important technique which could be applied to the surface of very soft deposits to provide a sufficiently trafficable surface such that conventional land farming techniques could be applied prior to revegetation activities. Alternatively, substantial amounts of fine tailings can be dewatered if thin layers are placed with allowance to freeze between each layer. This concept could be followed for several annual cycles within a mine pit. This chapter reviews research on the application of freeze-thaw dewatering and solids enhancement of Clarke Hot Water Extraction process (CHWE) fine tailings. The majority of the research has centered on freeze-thaw dewatering of mature fine tailings with solids contents of 30%. Research has included small scale laboratory freeze-thaw tests, large scale laboratory freeze-thaw tests and field freeze-thaw tests using large volumes of mature fine tails. Review of non-CHWE process fine tails freeze-thaw research is included in Volume IV.

4.1.1 Major Conclusions

(i) Laboratory freeze-thaw tests on fine tailings indicated dewatering can be improved significantly by chemical treatment of the fine tailings prior to freeze-thaw. Chemical treatment results in the production of a mineral-free runoff water whereas turbid water is released from untreated samples.

(ii) The hydraulic conductivity is significantly increased while compressibility is reduced by freeze-thaw of chemically treated fine tailings samples.

(iii) Results from the 1992/93 Suncor field tests showed that thin layer, freeze-thaw dewatering of acid/lime fine tailings is feasible resulting in ability to treat up to 3 times the volume of fine tailings per unit surface area; substantial volume reductions of 50 to 70% and significant increases in solids content from 35% to between 68 and 72% were observed. A mineral-free, easily detoxified runoff water was produced.

(iv) After a single summer season, natural evaporation had produced an extensive crust over the well drained areas of the test ponds which, along with the underlying fine tailings with undrained shear strength of 3 kPa, supported human traffic.

(v) The technical feasibility of these techniques has been established. Further efforts should concentrate on site specific economic feasibility assessments and construction of a full scale demonstration site.

4.2 Results from 1992/93 Suncor Field Tests

A large scale field evaluation of thin layered, freeze-thaw dewatering of Suncor acid/lime amended fine tailings was begun in 1992. Lowe (1993) documents the site facilities and operations during the 1992/93 winter in which three test ponds were filled with fine tailings. Figure 4-1 is plan drawing of the three ponds, 2A, 2B, and 2C, constructed adjacently to Suncor's Tailings Pond 2. Instrumentation was installed in Ponds 2A and 2B to monitor the settlement behavior of the fine tails during a cycle of freeze-thaw. Frozen cores were retrieved to provide samples for geotechnical testing and characterization. Further sampling and site characterization was performed later in 1993 as the material in the ponds thawed and consolidated.

A comparison of the initial and final solids contents and volume change which took place in the ponds during thaw, consolidation and desiccation are shown in Figure 4-2. The original frozen thickness of fine tailings in Ponds 2A and 2B were 1.60 m and 1.27 m, respectively. The processes of thaw, consolidation and surface drying (desiccation) reduced the heights of the fine tailings by between 50 and 70%. The average solids content of Pond 2A increased from 35% to 72% and for Pond 2B it increased from 35% to 68%. While laboratory freeze-thaw results in a solids concentration increase from 35% to approximately 44 to 48% (based on data from Figure 4-23), the additional mechanisms of consolidation and surface drainage and drying further increases the solids concentration to 70%. This solids content enhancement is evident in the change in solids contents with time in Ponds 2A and 2B as indicated in Figures 4-3 and 4-4 For example, Pond 2B increased its initial solids contents of 35% to 62% in June 1993, 62 to 68% by August 1993 and then remained at 68% by October 1993.

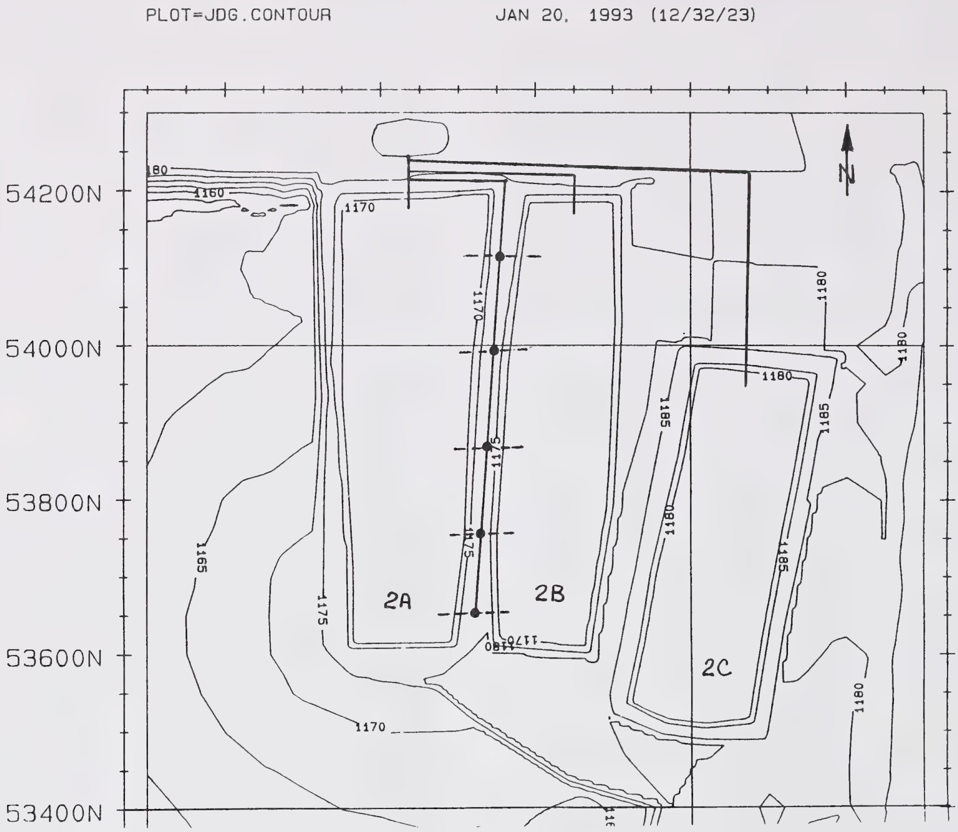


FIGURE 4-1. Plan view of Suncor freeze-thaw ponds.

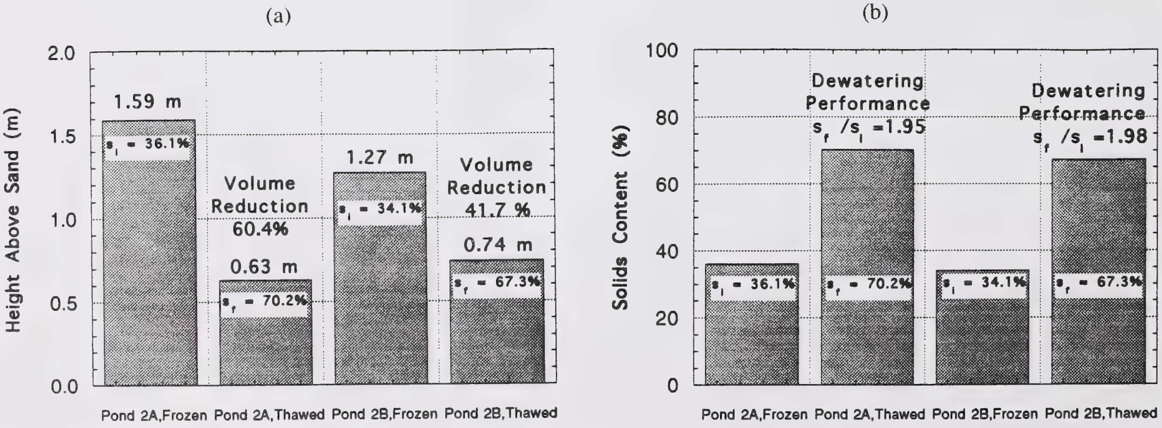


FIGURE 4-2. 1992/93 freeze-thaw field results (a) decrease in pond height and (b) increase in solids contents.

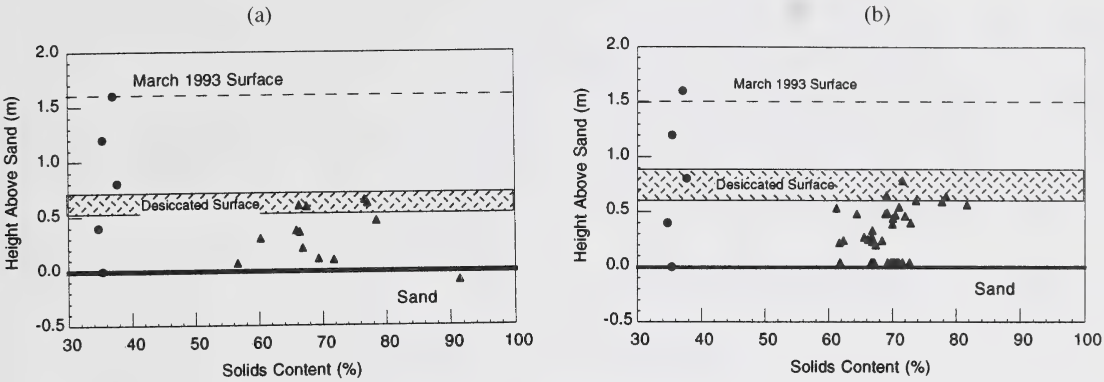

(a) (b)

FIGURE 4-3. Solids content with depth for Pond 2a, (a) 16 August 1993 and (b) 5 October 1993.

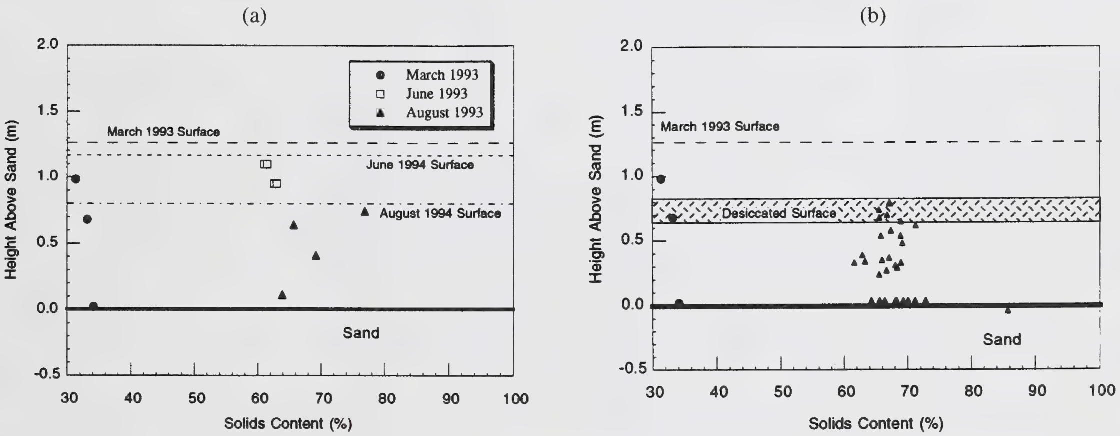

(a) (b)

FIGURE 4-4. Solids content with depth for Pond 2b, (a) June and August 1993 and (b) 5 October 1993.

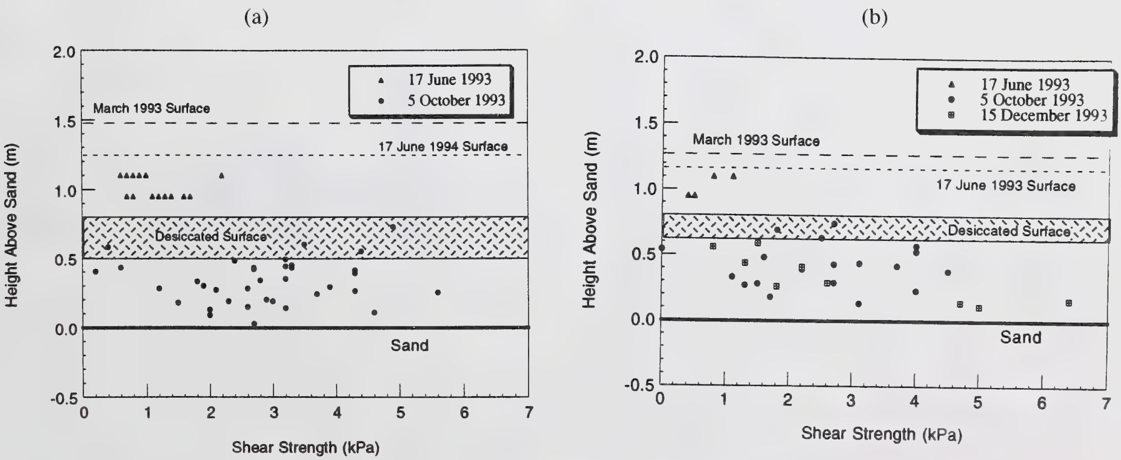

(a) (b)

FIGURE 4-5. Field vane undrained shear strength with depth (a) Pond 2a and (b) Pond 2b.

The change in solids content was also reflected by changes in the undrained shear strength as shown in Figure 4-5. From an initial value of 0.02 kPa for 35% solids fine tailings, the undrained shear strength increased to 1.5 kPa by June 1993 and underwent further enhancement to 3 kPa by October 1993. It is also apparent that the undrained shear strength is linked to the solids content as reflected by the higher strengths measured in the desiccated crust.

4.3 Initial Research on Freeze-Thaw Dewatering of Oil Sands Fine Tailings

4.3.1 Elliott's Patent

Elliott (1975) patented a process for releasing the clarified water from the middling fine tailings discharge associated with the Clarke hot water extraction process (CHWE). The water would then be suitable for recycling to the extraction process. In summary, the patented process involved the following steps: (i) agglomeration of the middlings fines (clay particles) through the addition of an agglomerating agent and modifying the solution pH, (ii) freezing the agglomerated middlings, (iii) thawing the frozen agglomerated middlings and (iv) separating the released clarified water from the thawed settled middlings fines. This process was to be carried out in settling pond zones and incorporate multiple cycles of freeze-thaw. Elliott claimed that the 6 to 12% suspended solids in the middlings could be increased to 30% solids by this process.

The freeze-thaw process is augmented by an agglomeration step which is accomplished by three possible methods: (i) by adding an agglomerating agent, such as sodium or calcium chlorides, alum, or calcium hydroxide; (ii) by adjusting the pH of the middlings below 7.5; or (iii) by adjusting the pH of the middlings above 9.0. Elliott's preferred method of agglomeration is accomplished by adding sulfuric acid (to the middlings or effluent discharge potion as the case may be) to about pH 5. He argues that this would alter the net charge on the edges of the clay particles and encourage agglomeration. His explanation is unclear but it may be based on an argument that a low pH condition allows the development of positive charges on the edges of the clay particles (van Olphen 1977). These positive charges would be attracted to the inherent negative charge on the faces of the clay particles. Edge to face associations would occur and this would develop a card house structure. Figure 4-6 shows the results of freeze-thaw tests on specimens with initial solids contents between 6 and 12% for four chemical amendments: $Al_2(SO_4)_3$, $FeCl_3$, $CaCl_2$, and H_2SO_4. The largest enhancement in solids content was achieved using H_2SO_4 in which a 6.5% solids middlings specimen increased to 26.6% solids (final to initial ratio of 4.09) and in which a 12.0% solids specimen increased to 22.2% (final to initial ratio of 2.32). His results indirectly show that freeze-thaw dewatering is sensitive to initial solids contents with the ratio of final to the initial solids contents decreasing as the initial solids content increases.

Additional cycles of freeze-thaw are claimed to eventually increase the solids content to 30%. Figure 4-7 shows the results of three multicycle freeze-thaw tests. Tests 1 and 2 were performed with 504 ppm of H_2SO_4 in which the solids content increased from 6.5% to 30% after two or three cycles. Test 3, performed with 13.7 ppm of $Al_2(SO_4)_3$, showed mixed results with no increase in solids content until the third freeze-thaw cycle.

These test results and the patent were intended for middlings fine tailings and not for mature fine tailings which had settled to 30% solid content in settling basins. However, the results indicated the potential for chemical addition to promote agglomeration and subsequent application of freeze-thaw to dewater fine tailings. Elliott also suggested alternate means for optimizing the freeze-thaw process. His suggestions included bottom pumping of unfrozen fine tailings onto the surface of frozen fine tailings to create a layered deposit and the pumping of warm water on the sur-

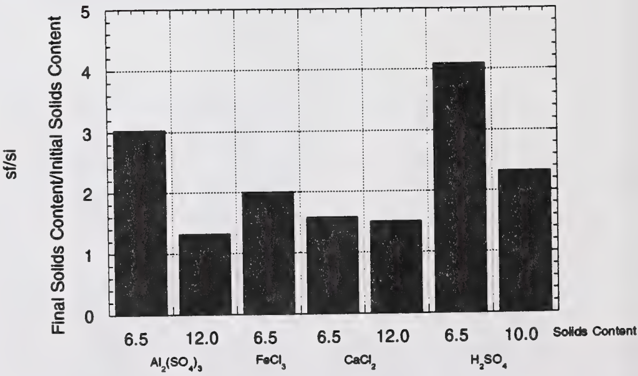


FIGURE 4-6. Comparison of chemical amendment on freeze-thaw dewatering of oil sands middlings from Elliott's patent.

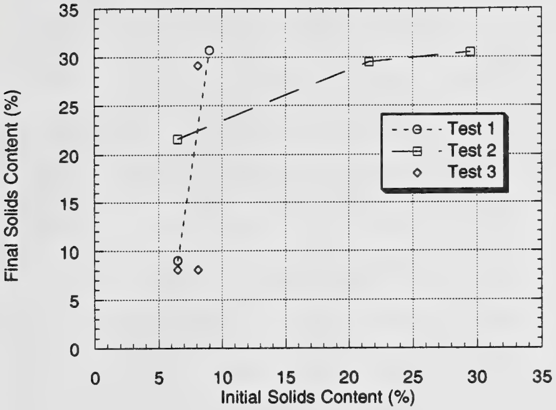


FIGURE 4-7. Multi-cycle freeze-thaw test results from Elliott's patent.

face of frozen fine tailings to accelerate the thawing process. Missing is consideration of the thermal and geotechnical mechanisms which could be engineered to maximize the volumes which could be treated by freeze-thaw.

4.3.2 Boyar's Patent

An alternate approach to freeze-thaw dewatering of oil sands fine tailings was patented by Boyar (1978) which covered a method for reducing the mineral (clay and silt) content of colloidal clay-water mixtures. The concept entails changing a clay-water suspension into droplets and expelling them into the atmosphere under freezing temperatures. The water would form snow-like crystals in a process which separates the water from the suspended solids. The resulting synthetic snow consists of an inner core of pure ice surrounded by a concentrated mixture of silt, clay and water. The snow settles onto to the ground to form a deposit. When the deposits melts during the following spring and summer, the melt water drains away and it no longer contains a colloidal dispersion of silt and clay. The melt water is collected and recycled while the deposit consists of a concentrated mixture of silt, clay and water (thawed settled solids). Boyar states that the process is most effective in the treatment of fine tailings with an initial solids content between 5 and 30%.

The patent provides an example of the process in which a 23% solids "aqueous sludge" at 16C is withdrawn from a retention pond. The sludge is passed through a heat exchanger to reduce its temperature to 1C. The chilled sludge is then pumped at 450 L/min at a pressure of 1000 kPa to a snow gun with a specified nozzle. Air is fed into the sludge at 1000 kPa at about 330 L/min. At an atmospheric temperature of –21C, an artificial snow is created from the sludge through the rapid expansion of the sludge-air mixture as it is expelled from the nozzle. Unfortunately Boyar provided no data to support his dewatering claims or to estimate the volume of clarified water and settled solids which remained in the deposit.

4.3.3 Research at the Alberta Environmental Centre (AEC)

Johnson *et al.* (1993) reports on research conducted over a six-year period at the Alberta Environmental Centre using Syncrude mature fine tailings to examine the dewatering *via* freeze-thaw and evapotranspiration. Their research program included small scale laboratory freeze thaw tests and model tests, large scale laboratory model tests, and three field experiments at AEC and on the Syncrude lease. They also examined the effect of evapotranspiration in removing the decant water from the thawed surface and enhancing the fine tailings solids contents. Various mixtures of sand-fine tails were also tested to investigate the effect of sand on the fine tailings water content and shear strength. The fine tails tested were from the Syncrude Mildred Lake Settling Basin and consisted of 25% solids with 47% clay (<2 um), 27% silt (2 to 50 um) and 26% sand (>50%).

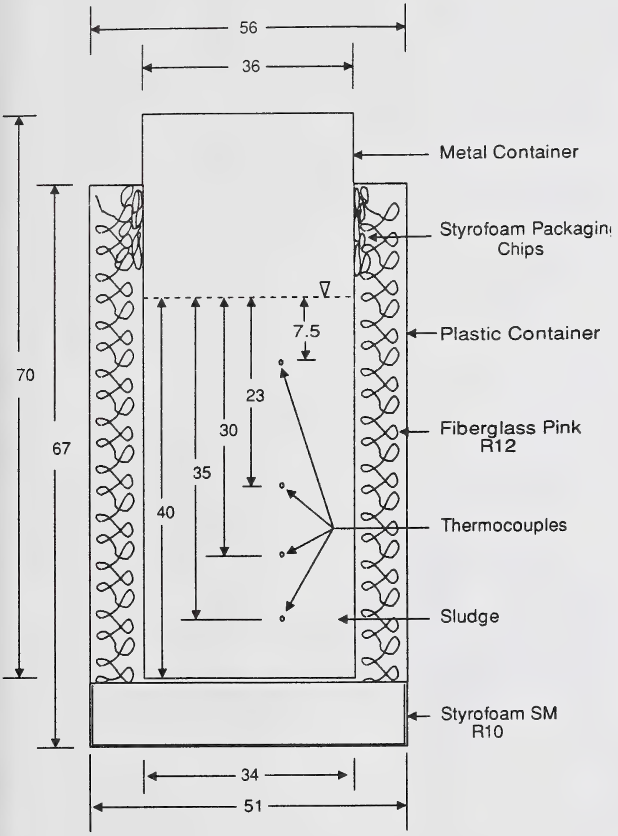
The assessment of freeze-thaw dewatering began with small scale laboratory tests. These tests consisted of 3 to 20 kg specimens frozen in containers placed either in a freezer at –24°C or outside during the winter. The specimens were subjected to 1 to 3 cycles of freeze-thaw. The tests showed that specimens with 29% to 35% initial solids contents increased to 52% to 54% solids content when thawed. To overcome the limitations of these three dimensional freezing tests with no drainage, an additional series of better controlled small scale model tests was performed. These experiments investigated various methods for enhancing drainage during the freeze-thaw dewatering process. Two box containers were used, one with a single vertical sand channel down the middle and the second with a sand liner along the bottom and sides. Fine tailings in these two experiments experienced increases in initial solids contents from 35% to 52% for the sand channel and up to 62% solids content for the sand liner. Finally a larger scale test was carried out in which "a child's plastic wading pool (0.9 m deep) was half-filled with damp sand and the other half was filled with 35% solids sludge" and it was placed outside to freeze. During thaw the water was absorbed by the sand resulting in a solids content of 56% in the settled fine tails and evaporation further increased the solids content of the fine tails to 75%.

Large scale laboratory model tests were performed to evaluate the Stefan-Neumann equation in predicting the advance of the frost depth into the fine tails. Figures 4-8 and 4-9 depict the apparatus used to carrying out these tests including the thermocouples for monitoring temperatures and the insulated double barrel design for producing one dimensional heat flow conditions similar to those found in nature. Freezing was conducted in a freezer at a temperature of –24°C. Four initial solids contents were evaluated, 15, 25, 35 and 45%, and the increase in solids contents as result of the freeze-thaw cycle are shown in Figure 4-10. These results show an exponential relationship between initial solids content and the increase in solids content upon thaw which suggests that the benefits of freeze-thaw are very sensitive to the initial solids content in the fine tails. Multilayer tests were also performed to evaluate the effect of multilayer freezing on subsequent dewatering and volume change in the fine tails. Four 114 mm thick layers were frozen in the test apparatus (Figure 4-9). Based on previous test results, 114 mm thick layers were estimated to require 24 hours to freeze completely. The multilayer test exhibited an increase in solids content from 25% to 46% with no vertical variation because of the layers. When comparing the single layer tests with the multilayer test the multilayer test achieved greater volume change (49% *versus* 44%) and greater increase in solids content (Figure 4-10). Several hypotheses were offered to explain the difference but no additional analyses or testing was performed to investigate the differences.

To evaluate the feasibility freeze-thaw dewatering technology at a field scale, three field tests were performed: (i) AEC test pits, (ii) Mildred Lake bin experiment and (iii) Mildred Lake pit experiment. The AEC test pits were 7 m long and 1.2 m wide and were filled to a depth of 1.5 m with 30% solids fine tailings. Temperature monitoring showed that the freezing front penetrated to a 1.00 m depth. Post thaw sampling gave solid contents of 47% in the upper 150 mm layer and 57% in the bottom 100 mm of the pit. The contents of the two pits were transferred to one pit and half the surface was seeded with reed canary grass. Both the unseeded surface and the seeded surface underwent significant dewatering during the summer. Figure 4-11 shows the variation in solids content with depth for the pit for post thaw, the unvegetated half and the vegetated half. The reed canary grass, which had established a complete canopy by Fall, increased the solids content from 50% to 70 to 88%. Evaporation in the unvegetated zone was nearly as effective with the final solids content between 0 and 10% less than recorded in the vegetated zone. Evaporation led to a well-developed 100 mm thick surface crust with measured solids contents of 88%.

A series of bin experiments was performed at the Mildred Lake site to (i) test the ability of plants to establish, grow and dewater both fine tailings and sand-fine tails mixtures, and (ii) test freeze-thaw dewatering on both fine tails and sand-fine tailings mixtures. Twelve grain bins 1.83 m high by 4.27 m in diameter with a volume of 25 m^3 were employed in the test program. Six bins were filled with fine tailings (solids content of 30%) or fine tailings-sand mixtures (1:3 ratio and solids content of 50 to 55%) and then planted with reed canary grass, western dock or left fallow as a control. Fertilizers were mixed with the waste materials and various surface treatments (for example, dry peat and mulch) were added to the surface. Observations suggested that it was difficult to establish plants directly on either the fine tailings or the fine tailings-sand mixture. Furthermore evapotranspiration was limited to the near surface and no significant increase in solids content was observed below the surface crust in either waste material. However when the bins were allowed to freeze completely during the following winter, there were significant increases in solids contents for both materials. The fine tails-sand mixture increased from 55% solids to 80% solids content through the entire depth and the fine tailings increased from 36% solids to 52 and 57% solids content by mid summer.

The Mildred Lake pit experiments were performed to overcome the size and thermal boundary condition limitations of the bin experiments. The objectives of the experiment were to evaluate dewatering under one dimensional freeze-thaw conditions, to evaluate ways of controlling surface drainage, and to develop a practical method for establishing plants on the thawed fine tailings surface. Two 60 m^2 × 2 m deep pits were excavated at Syncrude Canada Ltd.'s site at Mildred Lake (Figure 4-12) and lined with a 20 mil woven polyethylene geotextile to prevent drainage into the surrounding dykes. The engineered pits were designed with several measures to improve surface drainage and instrumentation to monitor performance during freeze-thaw. The pits were filled with one layer of fine tails (solids contents between 24 and 35%) and allowed to freeze during the winter. Since the pits were constructed on a 4-degree slope, the fine tailings depth varied across the width of the pit from 1.50 m to 0.25 m. Thermocouples monitored the temperature distribution through the pits and the measurements showed the fine tails completely froze. Table 4-1 provides two years of solids contents data for both pits at different locations and depths. After one year of freeze-thaw the fine tails had increased from 25–35% solids content to 41–53% solids content which improved to 55–63% after a second cycle of freeze-thaw. Surface drainage measurements were only partially successful and this affected the transplantation of vegetation. Reed canary grass survived and grew when the drainage was adequate while the western dock plants died soon after germinating on the surface.



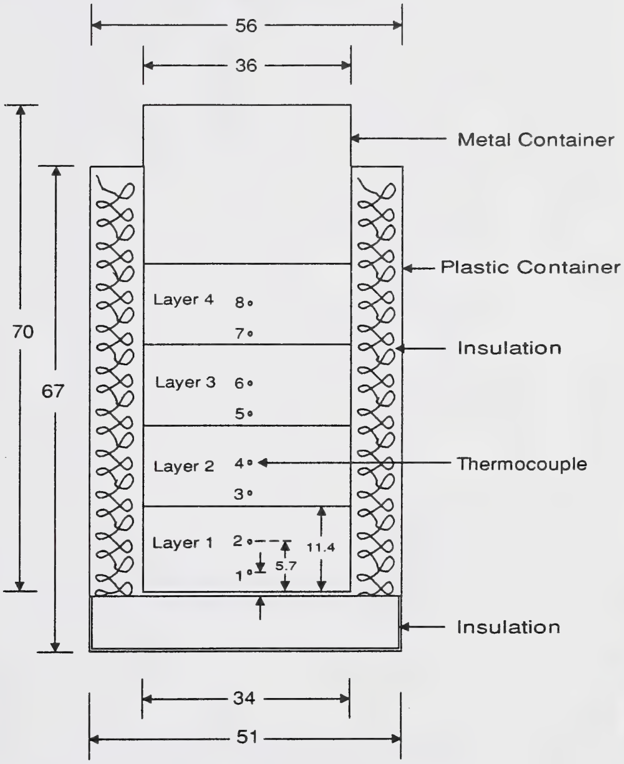


FIGURE 4-8. Instrumented insulated barrels used in Syncrude's laboratory freeze-thaw tests.

FIGURE 4-9. Layer configuration.

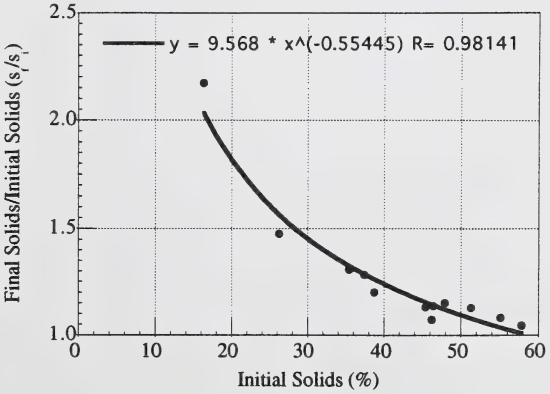


FIGURE 4-10. Change in solids content associated with freeze-thaw as a function of initial solids.

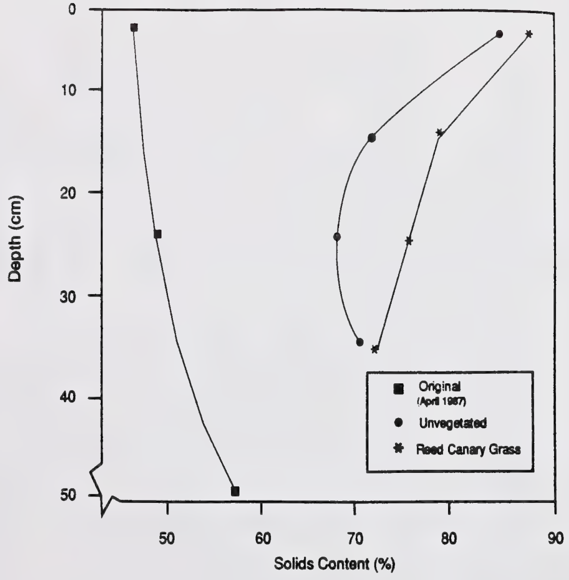


FIGURE 4-11. Effect of freeze-thaw and vegetation on solids content.

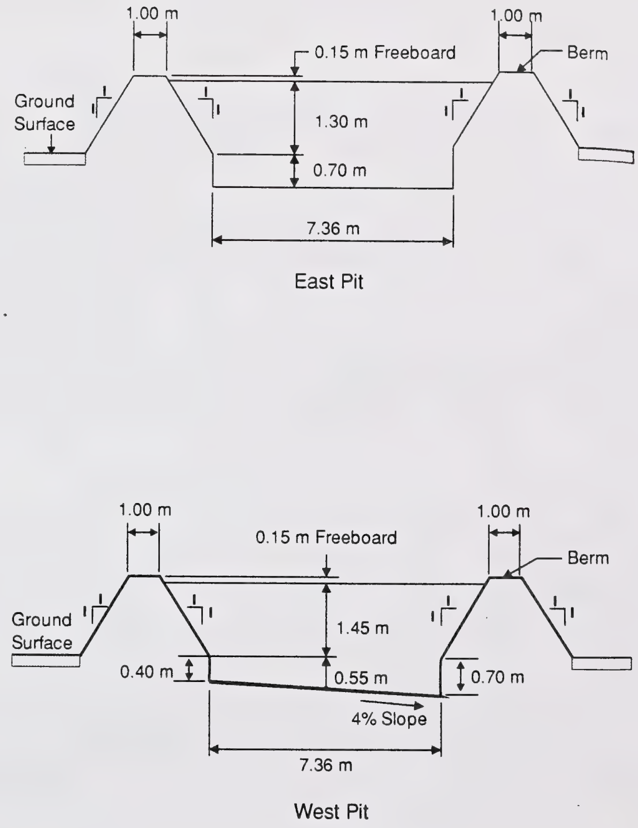


FIGURE 4-12. Cross-sections of east and west pits used in Syncrude freeze-thaw field experiments.

TABLE 4-1. Solids contents of Mildred Lake pit experiments (Johnson *et al.* 1993).

Depth (cm)	Pit	Sample date and solids contents (%)			
		Oct 1987[1]	July 1988	June 1989	Sept 1989[2]
10	West	28.4	52.7	54.9	57.6
	East	23.9	48.8	59.5	62.7
30	West	29.2	46.2	53.5	55.2
	East	24.9	47.4	53.0	61.6
50	West	30.8	46.1	51.2	55.0
	East	26.1	47.3	54.1	59.5
70	West	32.3	46.4	N/A[3]	60.5
	East	29.7	43.9	N/A	59.7
90	West	33.0	41.3	C[4]	C
	East	29.4	43.1	C	C
110	West	33.7	C	C	C
	East	28.9	C	C	C
150	West	32.4	C	C	C
	East	35.7	C	C	C

Notes: [1] October 1987 was the original sampling data immediately after the fine tailings were placed. [2] September 1987 was the final sampling date. [3] Not measured. [4] Since the fine tailings had consolidated considerably, the fine tailings pits were no longer deep enough to provide data at these depths.

4.4 University of Alberta Research

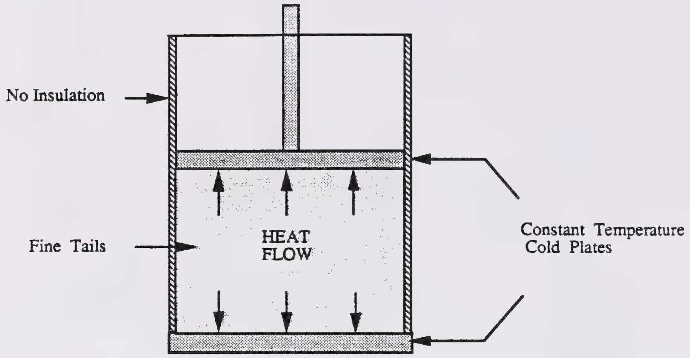
4.4.1 Laboratory Investigation of Freeze-Thaw Dewatering on Syncrude Fine Tailings

Research at the University of Alberta began with a program to evaluate the freeze-thaw dewatering and the post-thaw consolidation behavior of Syncrude fine tailings (Sego and Dawson 1992). Two sets of apparatus were built to investigate freeze-thaw dewatering (Figure 4-13). Single layer (thicknesses from 50 to 200 mm) freeze-thaw tests were performed in the conduction freezing apparatus which allowed an investigation of layer thicknesses and temperature boundary conditions on the solids content enhancement associated with cyclic freeze-thaw. The multilayer convection freezing apparatus provided testing of the thin layered freeze-thaw design concept. Subsequent post-thaw consolidation tests were conducted to detect the effects of the freeze-thaw cycle on the compressibility, hydraulic conductivity and consolidation behavior of the fine tails. The test material used was Syncrude mature fine tailings with a solids content between 28 and 30%, a bitumen content of 1%, 60% minerals finer than 2 μm, and liquid and the plastic limits were 55% and 25%, respectively.

Single layer conduction freeze-thaw tests were performed to determine the effects of layer thickness and temperature boundary conditions on solids enhancement. Freeze-thaw dewatering was assessed by comparing the initial specimen solids content (28–30%) with the thawed settled solids and decant water solids content. The results of these experiments are presented in Figure 4-14. Figures 4-14a and 4-14b indicate that as the layer thickness increases from 50 to 100, 150 and 250 mm, the thawed settled solids increases. The data in Figure 4-14c are less sensitive to layer thickness. When comparing temperature boundary conditions, the –8C at top and –15C at bottom boundary conditions (quickest freezing), produced thawed settled solids contents of 35 to 38% as compared to 38 to 44% for the other two boundary conditions. Both the layer thickness data and the boundary condition data imply that lower freezing rates result in higher thawed settled solids contents.

A multicycle freeze-thaw test was performed on a 150 mm thick layer under –8 and –15C temperature boundary conditions (Figure 4-15). During the first cycle the initial solid content increased from 28 to 34%. After second and third cycles the settled solids increased to 43% and 45%, respectively. These results show that freeze-thaw dewatering

A. Single Layer Conduction Freezing Apparatus



B. Multi-layer Convection Freezing Apparatus

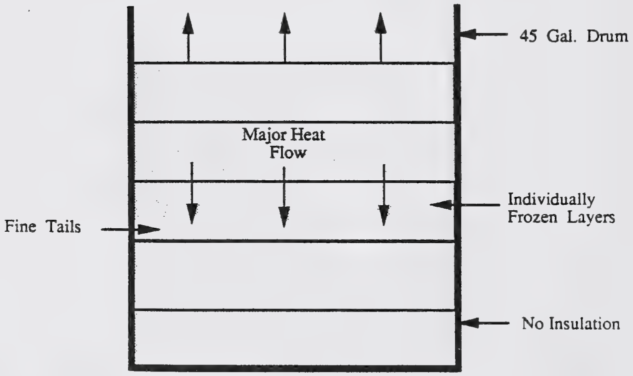


FIGURE 4-13. University of Alberta's laboratory freeze-thaw apparatus (a) single layer conduction and (b) multi-layer convection.

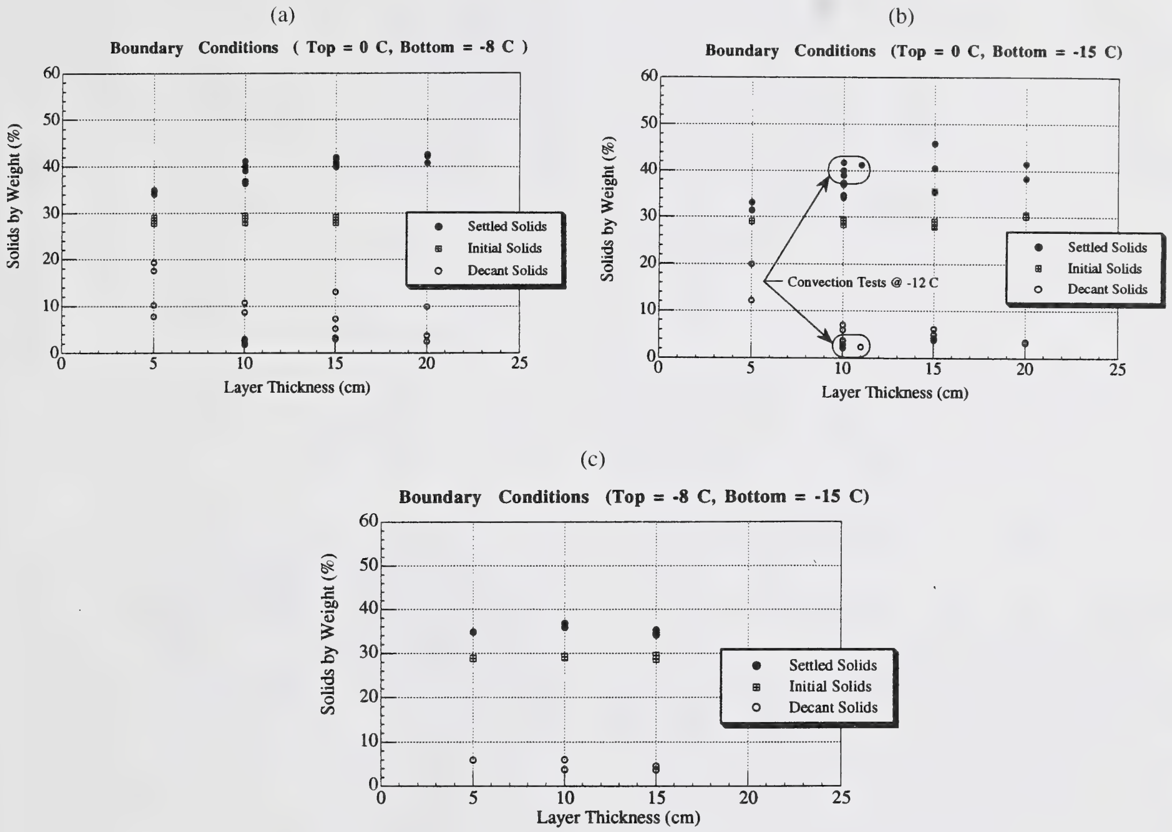


FIGURE 4-14. Single layer conduction freeze-thaw test results.

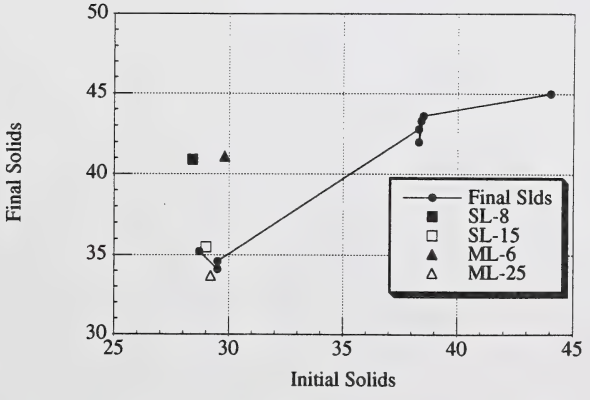


FIGURE 4-15. Multi-cycle freeze-thaw results for 150 mm layer with –8°C and –15°C boundary conditions.

has a reduced effectiveness after the second cycle of freeze-thaw. This was confirmed by the undrained shear strength results which increased significantly in the first two cycles of freeze-thaw but was less effective in the third cycle.

Three multilayer convection freeze-thaw tests were performed with the apparatus shown in Figure 4-14b. Table 4-2 summarizes the results for (i) fully frozen at –6C (with additional cycles), (ii) full frozen at –25C (two initial solids contents), and (iii) partially frozen at –6C. Figure 4-15 allows a comparison of the single layer and multilayer freeze-thaw tests. The –8C single layer test and the –6C multilayer test performance are similar and result in significantly greater solids enhancement than the –15C and –8C/–15C single layer tests or the –25C multilayer test. From these results it is evident that the freezing temperature has a significant effect on solids enhancement. The –6C test showed a 37.9% increase in solids content as compared to the –25C test which showed only a 15.4% increase in solids content. The variation in solids content with depth for the thawed –6C multilayered test is shown in Figure 4-16. The decant water contains 2 to 3% solids, a transition zone between the decant and settled solids occurs and the settled solids averages around 41% solids content.

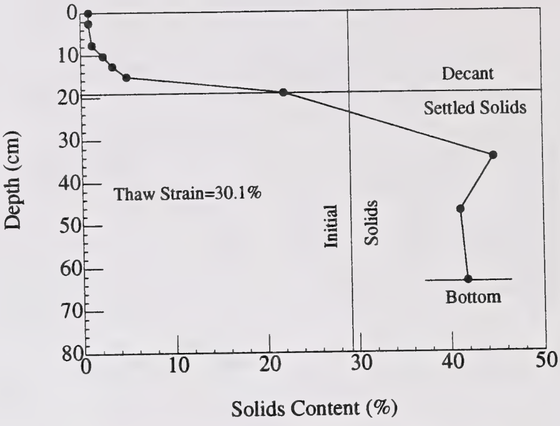


FIGURE 4-16. Multi-layer solids contents results.

The compressibility and hydraulic conductivity of the never frozen fine tailings and thawed settled fine tails were measured using a constant rate-of-deformation (CRD), one dimensional compression apparatus. The CRD tests have certain advantages over the conventional step loading compression test apparatus for low solids content materials such as fine tailings (Sego and Dawson 1992). The compressibility is obtained from a plot of void ratio *versus* effective stress and the results for fine tails and thawed settled solids (frozen in a 150 mm layer at –6C) are presented in Figure 4-17. The curves converge at a void ratio of 4 and an effective stress of 2 to 4 kPa. Sego and Dawson (1992) suggested that this effective stress represents the strength limit of the soil peds (masses of compressed soil about 2 mm in diameter) created during the freezing process. At higher stresses the peds break down and the thawed fine tailings responds as if it were never frozen. The hydraulic conductivity data in Figure 4-18 suggests that fine tails and thawed fine tails have hydraulic conductivities similar to that of clay soils. Because of the scatter in these data, no conclusions were made regarding the comparative behavior.

TABLE 4-2. Multilayer freeze-thaw test summary (Sego and Dawson 1992).

Test	Test conditions	Solids content (%)			Ratio of thawed to initial	Thaw strain (%)
		Initial	Decant	Thawed		
A	–6°C fully frozen	29.8	3.4	41.1	1.38	30.1
	–6°C partially frozen					
B	1st freezing	29.0	10.8	32.9	1.13	17.8
C	2nd freezing	29.0	6.9	34.7	1.20	20.3
D	3rd freezing	29.0	4.0	37.7	1.30	22.5
	–25°C fully frozen					
E	Low solids	29.2	16.0	33.7	1.15	25.5
F	High solids	31.6	7.5	36.6	1.16	16.7

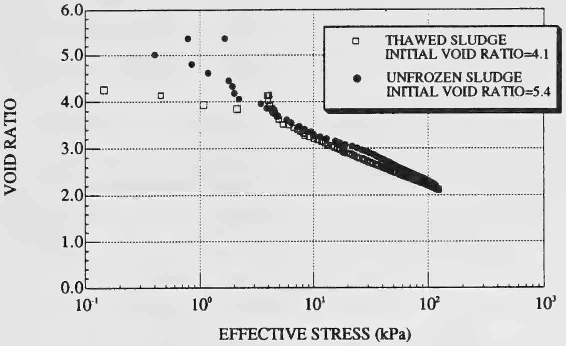


FIGURE 4-17. Compressibility of freeze-thaw fine tailings and never frozen fine tailings

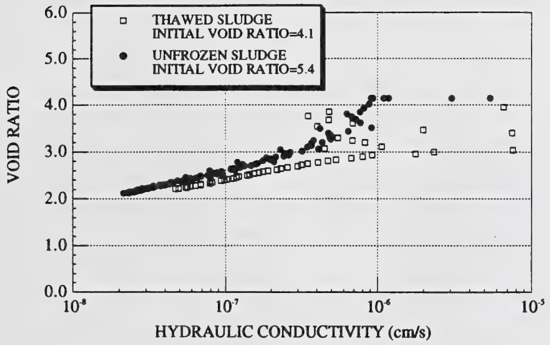


FIGURE 4-18. Hydraulic conductivity of freeze-thaw fine tailings and never frozen fine tailings.

4.4.2 Laboratory Investigation of Freeze-Thaw Dewatering of Suncor Fine Tails

4.4.2.1 *Freeze-Thaw Tests*

Sego (1992) reported the results of laboratory study on freeze-thaw tests to dewater thin layers of as received and chemically amended Suncor fine tailings. The closed system freeze-thaw apparatus developed by Sego and Dawson (1992) was employed in this study. Single layer specimens of 150 mm high by 100 mm diameter were frozen with a top and bottom cold plate temperatures of –8C and –15C, respectively. For the tests on as received fine tails, Suncor fine tails with a solids content of 30.3%, bitumen content of 1%, and clay fraction (<2 μm) of 49% was tested. Figure 4-19 is a plot of initial solids contents *versus* thawed settled solids contents for the Suncor fine tails and OSLO cold water extraction (OCWE) process fine tailings (Sego and Dawson 1992a) is provided for comparison. After one cycle of freeze-thaw the solids content of the Suncor fine tails increases from 30% to 40% and increases to 44% and 50% for second and third cycles. The OCWE process fine tails, which do not employ caustic (NaOH) in the bitumen extraction process, performed much better.

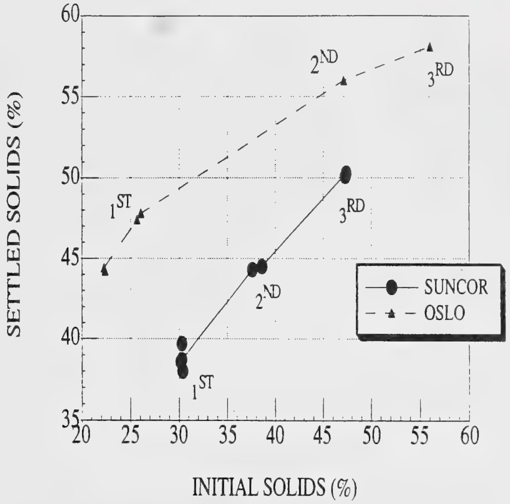


FIGURE 4-19. Multi-cycle freeze-thaw test results for Suncor and OSLO.

Based on this comparison of the Suncor and OCWE fine tails freeze-thaw results, it was concluded that the Suncor fine tailings performance could be enhanced through chemical amendment before freezing. First, the lowering of the pH if the fine tails pore fluid by the addition of sulfuric acid was evaluated. Due to the caustic extraction process employed to remove the bitumen, fine tailings are originally alkaline with a pH of about 8. Subsequently, the effect of adding flocculating agents to the pH altered fine tails was examined. As shown in Figure 4-20, for a lime amendment at 413 ppm, optimal thawed settled solids occurs at a pH of 6. In terms of the lime amendment concentration, it was observed that the optimum thawed settled solids contents occurred at a concentration of 413 ppm as shown in Figure 4-21. In comparison to as received fine tails with thaw settled solids of 40%, the acid and lime amended fine tailings had higher thawed solids contents between 43 and 46%. These freeze-thaw tests also showed that decant released upon thaw had lower solids contents.

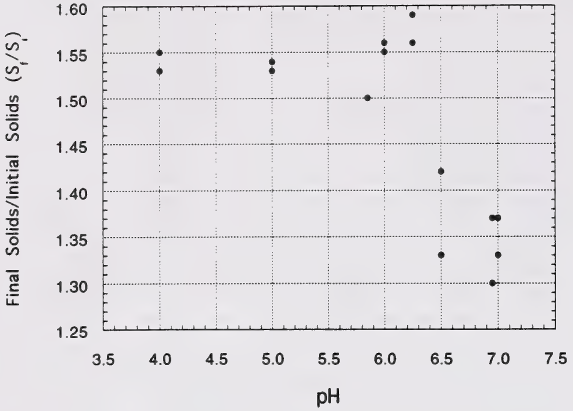


FIGURE 4-20. Effect of pH on freeze-thaw solids content.

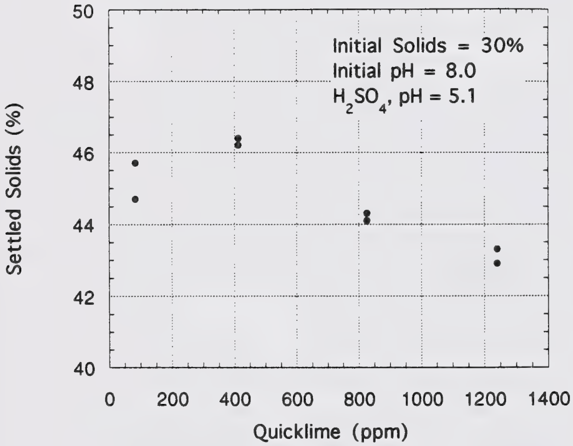


FIGURE 4-21. Effect of quicklime amendment concentration on freeze-thaw solids content.

4.4.2.2 *Post Thaw Consolidation Behavior of Suncor Fine Tails*

Sego and Proskin (1993) report the results of a geotechnical laboratory program to determine the post-thaw consolidation behavior of both as received Suncor fine tails and acid/lime amended fine tails. A large strain oedometer cell modified to run constant head permeability tests was employed to determine the consolidation, compressibility and hydraulic conductivity properties of the fine tails. The mineralogy and pore fluid chemistry of the Suncor fine tailings specimens were also evaluated.

As reported in 4.4.1 Sego and Dawson (1992) evaluated the consolidation behavior of the as received and once frozen Syncrude fine tailings with a constant rate of strain compression apparatus to avoid many of the limitations of step loading tests. However such tests are more difficult to perform and interpretation of the test results proved to be troublesome due to the development of a surface crust with lower permeability during the test. A large strain oedometer cell was therefore developed to accommodate the large strains (40%) and it uses standard geotechnical testing techniques for determining the consolidation and compressibility properties of soils. The cell was also modified to allow constant permeability head tests to be performed to independently evaluate the hydraulic conductivity of the fine tails as it is consolidated under various effective stresses.

In these tests frozen specimens were installed in the cells and allowed to thaw before applying the first load. After the specimen was consolidated under each effective stress (0.5, 2, 4, 10, 20, 50 and 100 kPa), a constant head was applied across the specimen, the outflow rate measured, and the hydraulic conductivity calculated based on Darcy's Law.

The compressibility curves for both the as received and acid/lime amended, once frozen and thawed Suncor fine tails are present as void ratio *versus* effective stress in Figure 4-22. Due to variations in initial void ratio and amounts of strain upon thaw, the curves begin at different initial void ratios. Both curves have a slight virgin compression curve before becoming linear at effective stresses exceeding 10 kPa and approach a void ratio of 1 at the final effective stress of 100 kPa. Comparison of the void ratio *versus* hydraulic conductivity curves in Figure 4-22b suggests that the chemically amended hydraulic conductivity was 10 times higher at high void ratios but converges to the as received curves at lower void ratios (and higher effective stresses). Based on these data it is concluded that acid/lime amendment, through its higher dewatering, lowers the initial void ratio for the compressibility curve and it increases the hydraulic conductivity but its influence is reduced at higher applied effective stresses and void ratios below about 1.

4.4.3 Comparison of Oil Sands Fine Tails Freeze-Thaw Behavior

A detailed comparison of Clarke Hot Water Extraction (CHWE) process fine tailings freeze-thaw dewatering behavior was reported by Sego (1994). Three sources of CHWE fine tailings, Syncrude 1992, 1993 and Suncor, were evaluated. The dewatering behavior of the as received fine tailings is compared in Figure 4-23 which shows the variation in dewatering behavior. By examining the index properties of

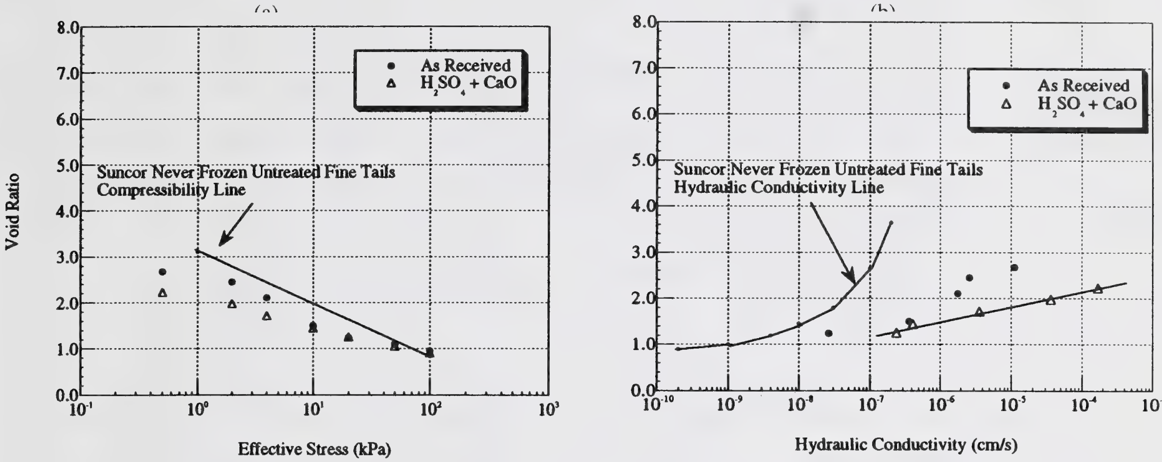


FIGURE 4-22. (a) Compressibility and (b) hydraulic conductivity results for untreated and treated Suncor fine tailings.

the three fine tailings, including solids content, bitumen content, per cent clay (< 2 μm), it was evident that the different fine tails were not homogeneous and freeze-thaw response was affected by these variations. Chemical amendment to reduce pH to 6 by adding H_2SO_4 followed by the addition of 413 ppm lime was carried out on the Suncor and Syncrude 1992 fine tailings. The freeze-thaw dewatering results are compiled in Figure 4-24 which suggest that both Suncor and Syncrude fine tailings experience significantly higher thawed settled solids following chemical amendment.

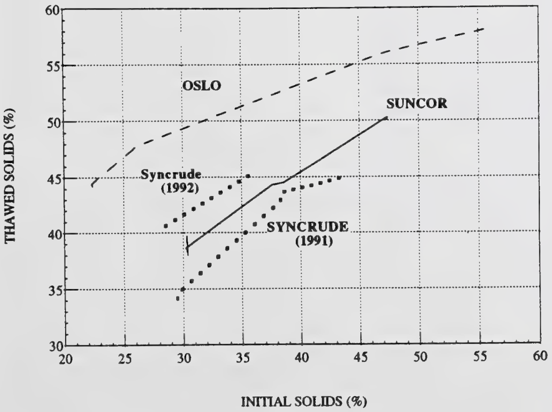


FIGURE 4-23. Comparison of freeze-thaw solids enhancement of as received fine tails.

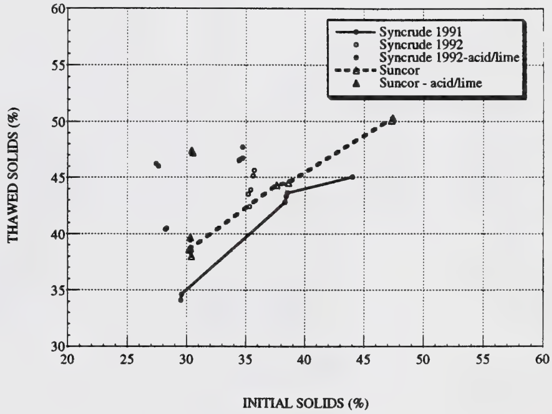


FIGURE 4-24. Freeze-thaw solids enhancement of chemically amended Syncrude fine tailings.

To evaluate the post-thaw dewatering behavior, consolidation and permeability tests were performed. Step loading oedometer tests were performed to evaluate the compressibility and hydraulic conductivity behavior of the fine tails. In Figure 4-25 the frozen-thawed Suncor and Syncrude fine tailings compressibility and hydraulic conductivity data are presented. The fine tailings compressibility is very close and converges with the never frozen fine tails data at void ratio of 1. In terms of hydraulic conductivity, both Suncor and Syncrude have higher values

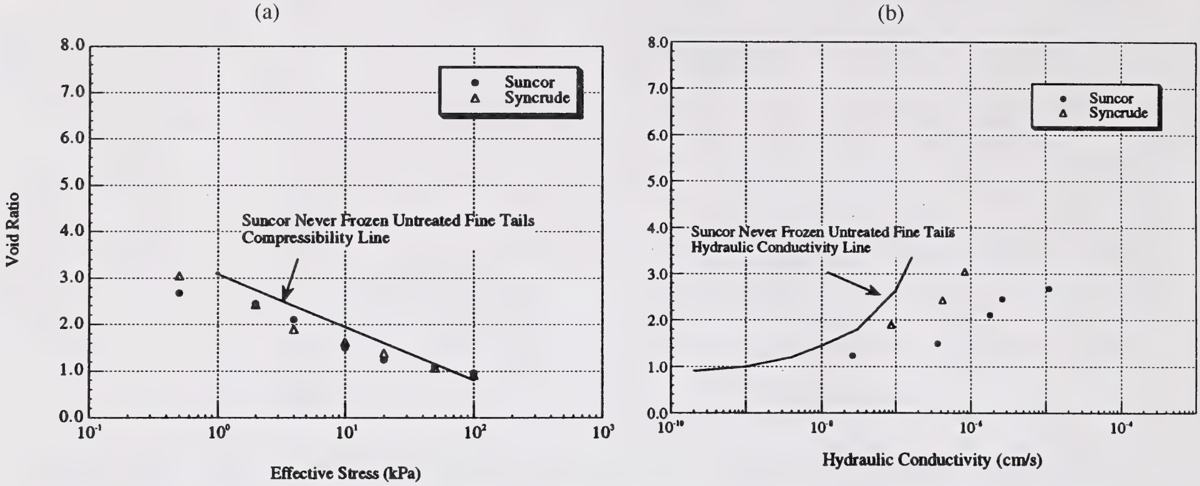


FIGURE 4-25. (a) Compressibility and (b) hydraulic conductivity of Suncor and Syncrude freeze-thaw fine tailings.

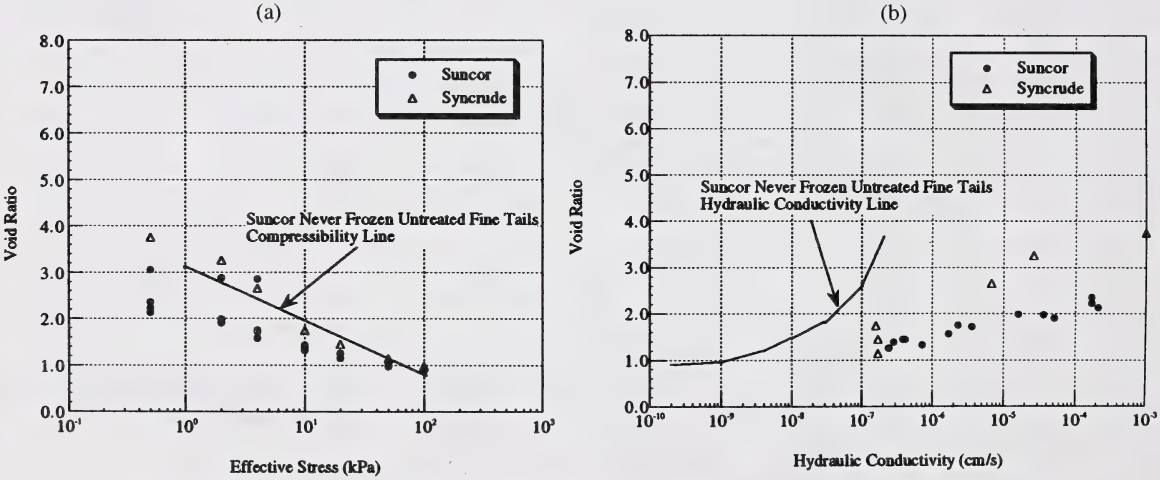


FIGURE 4-26. (a) Compressibility and (b) hydraulic conductivity of Suncor and Syncrude treated freeze-thaw fine tailings.

than the never frozen fine tails data for a given void ratio (effective stress). However, the Syncrude data appears to be almost one order of magnitude greater than the Suncor data. Similar observations were made for the chemical amended Suncor and Syncrude frozen and thawed fine tails (Figure 4-26). Compressibility behavior may be less affected by chemical amendment while hydraulic conductivity is significantly increased as compared with the as received fine tails. As with the as received data, the chemical amended Syncrude fine tails are about one order of magnitude more permeable than the Suncor fine tails for the same void ratio.

4.5 References

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5.0 USE OF NST RELEASE WATER FOR RECYCLE TO EXTRACTION

5.1 Introduction

Implementation of nonsegregating tailings (see Chapter 3.0 for a discussion of this process) will release water with a different chemical composition from normal tailings water. This water must either be stored in especially constructed ponds, recycled to extraction as process makeup, or discharged to the environment. Storage of this water represents little advantage to commercial operators over storing MFT. Preliminary investigations have determined that discharge of the NST release water to the river is not precluded based on the water chemistry or toxicity, as will be discussed in Chapter 6.0. The option of using this water as makeup for extraction processing is discussed in this chapter. Recent work has established that recycling of the NST release water to extraction is possible if calcium levels do not exceed the 3 mMole/litre level where bitumen recovery could be affected [1].

5.2 NST Release Water Chemistry

To create a stable NST mixture with a 3.5:1 sand to fines ratio, with 55% (by weight) solids content, the addition of 600ppm agricultural grade gypsum (85% $CaSO_4 \cdot 2H_2O$) was experimentally determined to be a reasonable operating concentration to prevent segregation [2]. (This is equivalent to 900 grams of calcium sulphate per cubic meter of tailings mixture.) Table 5-1 summarizes the concentrations of significant ions in NST release water based on assuming there is no recycling of release water. Data for river water and recycle water is also included. At the anticipated treatment level, there is only 1.2 mMole/litre calcium in the release water; which is below that which should cause problems with extraction recovery.

TABLE 5-1. Major ions in recycle water, river water, and NST release water. The elevated bicarbonate concentration in NST release water relative to river water is for NST created by gypsum addition. These numbers are typical for river water and recycle water and for NST with 55% solids and 3.5:1 sand to fines ratio created with 600 ppm agricultural grade gypsum.

	Ca^{2+}	HCO_3^-	SO_4^{2-}	Mg^{2+}	Na^+
Recycle water	0.17	14.2	0.94	0.16	16.1
River water	0.63	2.0	0.21	0.39	0.77
NST release	1.18	10.3	13.2	0.86	23.3

5.3 Impact on Recycle Water Chemistry

Blending NST release water into the recycle water pool will initially result in further dilution, thereby bringing the calcium concentration even lower. However, most of the process water is repeatedly recycled leading to the potential for build up of concentration. In order to quantify the calcium build up over time, the sources and sinks of calcium must be understood. These are shown in Figure 5-1. Table 5-2 illustrates two important sources of bicarbonate in oil sands processing: connate water in the oil sands itself, and absorption of carbon dioxide from the air at elevated pH. Two major sinks for calcium are precipitation as calcium carbonate, and absorption on, or near, clay surfaces. An additional sink for calcium is in the recycle water-filled voids of sand dykes and beaches constructed in areas of the operation which are not part of the nonsegregating tailings disposal process. Water lost to these voids in replaced from the River, implying that a maximum concentration of conservative ions can be estimated for long times into the future based on a site specific projected water balance.

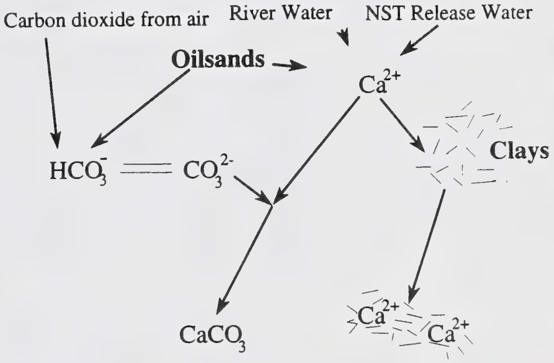

FIGURE 5-1. Schematic of sources and sinks for bicarbonate and calcium. All of the arrows represent equilibria.

Figure 5-2 shows how calcium and bicarbonate concentration change experimentally with increased calcium loading on a fresh tailings sample. The measured calcium concentration is well below the added concentration due to adsorption at the clay surfaces. After the clays are saturated, the bicarbonate concentration begins to drop as calcium is taken out of solution by precipitation as calcium carbonate [1]. When calcium is present in excess, magnesium concentration in solution increases as the clay adsorption

TABLE 5-2. Sources of bicarbonate. The increases observed with aeration at 80°C with time and from the oil sands itself.

	Total carbonate increase (mMole/litre)
Low grade oil sand (1100 g in 4.5 litres)	0.3
High grade oil sand (1100 g in 4.5 litres)	0.3
Aerated water (pH 10, 30min)	1.3
Aerated water (pH 10, 4 hours)	5.1

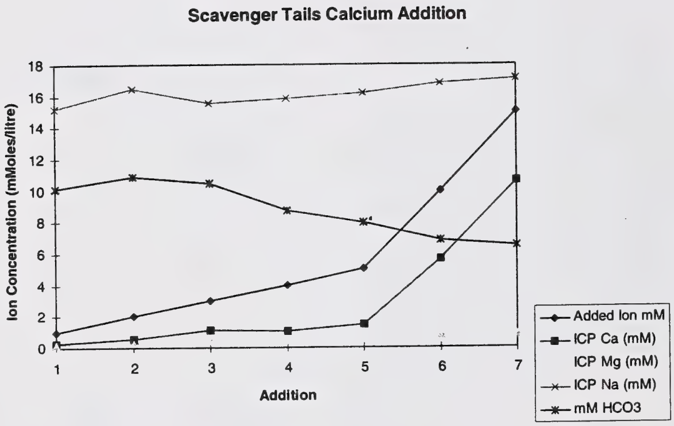

FIGURE 5-2. Calcium, bicarbonate, and magnesium concentration compared to added calcium concentration. The difference between the measured and added is the sink for calcium on the clay surfaces. Once the clays are saturated, bicarbonate decreases as excess calcium precipitates as calcium carbonate.

equilibrium is shifted by the increased calcium. The decrease in bicarbonate concentration shown in Figure 5-2 corresponds to the calcium concentration where bitumen recovery begins to deteriorate (see Figure 5-3).

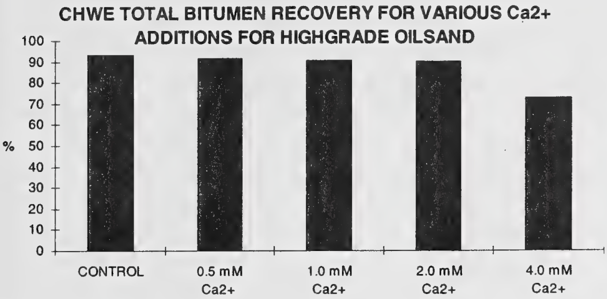

FIGURE 5-3. Bitumen recovery as a function of calcium concentration in the extraction water. Recovery deteriorates corresponding to the bicarbonate decrease in Figure 5-2.

5.4 Projected Impact on Extraction Process Performance

For the Suncor's current production rates and ore fines concentration, it has been determined that the clay surfaces can potentially act as a "sink" for the added NST calcium on the order of 900 t/year. The calcium loading expected from the creation of NST is only about 800 t/year. Therefore, it is anticipated that NST release water combined with the recycle water will have no detrimental impact on extraction. Over the long term, the only significant consequence (in terms of water chemistry) of the NST tailings treatment option may be elevated levels of sulphate [1].

As a replacement for river water, the NST release water has only twice the calcium concentration. The use of NST release water (from NST mixtures created with gypsum) for make up to extraction has an advantage over river water because of the increased bicarbonate concentration in the NST release water. This means a lower rate of caustic consumption will be required compared with bringing in new water from the River. It should be noted that when NST is created using acid-lime treatment, the advantage of maintaining the bicarbonate at high levels is lost.

5.5 Enhancement of Fines Settling

The fines require up to three months to flocculate and settle in the tailings pond to form fine tailings. It takes about three years to form MFT. It has been shown that increased calcium concentration enhances settling of the fine tailings in the recycle water ponds (Figure 5-4). The implication is that much less pond settling area will be required to reduce the mineral concentration to levels compatible with extraction makeup water requirements.

5.6 Future Developmental Activities

Syncrude is to conduct the first pilot test program using gypsum treatment in 1995. Suncor is anticipating a Commercial Trial test program involving utilization of about 50% of the total tailings produced over a six months period. This Trial is scheduled to commence in late 1995.

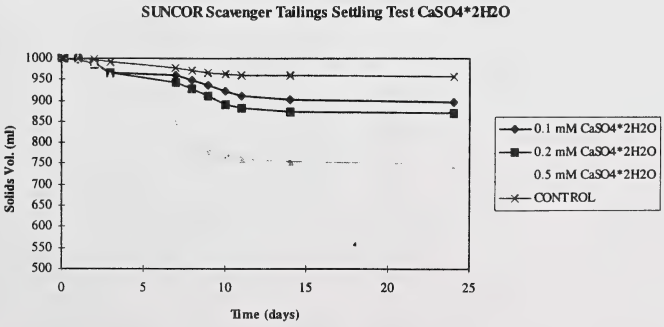

FIGURE 5-4. Scavenger tailings settling as a function of calcium concentration from 0.1 to 0.5 mMoles/litre. This is the likely range of concentration that would be encountered after dilution of the NST release water with the fine tailings stream.

When Suncor's coke fired boiler and flue gas scrubber begins operation in mid-1996, it will provide a ready source of gypsum as a waste product. This gypsum can potentially be used to create NST thereby displacing other commercial gypsum sources while yielding significant cost savings. The overall chemistry of this waste stream will not be known until the unit is operation, although it is anticipated that it will be compatible with NST requirements.

Further work is underway to determine the impact, if any, of the elevated sulphate levels that will result with the use of NST release water for recycling to extraction, and on the impact of impurities in the flue gas scrubber solids on NST release water chemistry.

5.7 References

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2. Scott, D.S., personal communication.

6.0 ENVIRONMENTAL DISCHARGE OF NST RELEASE WATER

As discussed in Chapter 5.0, the implementation of non-segregating tailings (NST) as a reclamation option will initially result in an increase in the water inventory. An option in dealing with this excess inventory is to treat the water and discharge to the environment. Recent studies have been done to determine the feasibility of this option. One study involved the sampling of release water from NST field and laboratory tests for complete chemical and toxicological analysis [1].

Initially, release water from NST treated tailings shows a certain level of acute toxicity, but with time, the acute toxicity level decreases. When the release water was decanted from the solids, the water was found to have no acute toxicity after a period of storage time (as determined using trout LC50, and daphnia LC_{50} assays, Table 6-1). As has already been determined from prior studies of oil sands tailings water, there are natural detoxification processes that occur [2,3]. The yet to be resolved question is the level of chronic toxicity (if any) that exists. This research is currently underway.

Another remaining question deals with the impact of the chemical makeup of the release water on the ecology of the receiving systems. The inorganic and organic compositions of NST release water were compared to existing river concentrations to evaluate this effect [4]. Table 6-2 contains some data on the water chemistry of two NST release water samples with those for the Athabasca River. Most of the river water values reported here are the average of the median values taken over four seasons and the maximum observed during the study year [5]. The NST values are from samples taken in June 1994 from a Suncor NST field trial [1]. The resultant concentrations of the components in the river were calculated based on a discharge rate of 5 m3/s and the winter 7Q10 flow rate for the river (a smaller value than this minimum 7-day flow rate would only occur once in 10 years) and then compared with actual river values and to current guidelines for surface water quality [4].

The decanted NST release water met tentative discharge and post discharge surface water criteria while the non-decanted water only failed to meet the pH requirement. Two parameters may be exceptions, total ammoniacal nitrogen and the chemical oxygen demand, but both were reduced in NST water samples as compared with untreated samples [4].

In order to bring NST release water to discharge criteria, current treatment options being evaluated include an initial, passive, biological detoxification of the water in holding ponds, after which the release water would be decanted to another pond for a season. Once the water met discharge requirements, it could then be discharged through natural wetlands to the environment.

TABLE 1. Results of toxicity tests of various NST release water samples. The Suncor data are from one set of samples taken in June, 1994 from field tests started in 1993. The CANMET data are from samples taken in November 1994 (decanted in October) from tests started in August 1994.

Sample		Trout LC_{50} (%)	Bacterial luminescence[a] (% of control)
Suncor field test — decant water	pit 1	>100	98
"	pit 2	N.M.[b]	100
"	pit 3	N.M.	87
Suncor field test — not-decanted	pit 1	N.M.	57
"	pit 2	N.M.	70
"	pit 3	N.M.	84
"	pit 4	N.M.	72
CANMET test	pool 1	71	91
"	pool 2	71	73

[a] The closer the value is to 100%, the lower the acute toxicity.

[b] Not measured.

TABLE 6-2. Inorganic and organic parameters for decanted NST release water and undecanted NST release water as compared to Athabasca River values.

Assay values	Athabasca River median value	Athabasca River maximum value	NST decanted water	NST undecanted water
pH	7.8	8.4	8.2	8.7
conductivity (mS/cm)	0.263	0.369	2.180	2.260
alkalinity (mg/L)	114	164	296	ND
hardness as $CaCO_3$ (mg/L)	122	183	200	213
Ca (mg/L)	34	51	51	41
Mg (mg/L)	9	14	20	27
Na (mg/L)	8	16	456	474
K (mg/L)	1.0	3.6	11.7	19.3
Al (mg/L)	0.54	11.40	0.08	0.74
As (mg/L)	0.0008	0.0210	<DL[a]	<DL
Ba (mg/L)	ND[b]	0.086[c]	0.020	0.024
B (mg/L)	0.053	0.150	1.000	3.100
Cd (mg/L)	ND	0.0018[c]	<DL	<DL
Cr (mg/L)	ND	0.005[c]	<DL	<DL
Cu (mg/L)	0.003	0.040	0.006	<DL
Fe (mg/L)	1.67	28.00	0.06	0.11
Pb (mg/L)	N.D.	0.005[c]	<DL	0.04
Mn (mg/L)	0.08	0.74	0.02	0.01
Hg (mg/L)	N.D.	0.00004[c]	0.0001	ND
Mo (mg/L)	N.D.	0.0034[c]	0.13	0.24
Ni (mg/L)	0.004	0.035	0.014	<DL
P (mg/L)	0.095	2.500	<DL	<DL
Se (mg/L)	N.D.	<DL[c]	<DL	<DL
Si (mg/L)	2.9	4.9	4.10	6.20
V (mg/L)	0.002	0.015	0.009	<DL
Zn (mg/L)	0.007	0.120	0.081	0.042
HCO_3 (mg/L)	137	200	357	244
Cl (mg/L)	3	5	59	64
SO_4 (mg/L)	23	37	932	1044
PO_4 (mg/L)	0.017	0.044	<DL	<DL
NO_3/NO_2 (mg/L)	0.05	0.42	0.08	<DL
F (mg/L)	ND	0.13[c]	12	17
CN (mg/L)	0.004	0.021	<DL	ND
TOC (mg/L)	9	30	52	59
oil + grease (mg/L)	0.7	3.3	1.1	ND
phenols (mg/L)	0.003	0.041	0.006	ND
BOD (mg/L)	ND	25[c]	3	ND
COD (mg/L)	ND	21[c]	134	ND

[a] Less than detection limit; [b] No data available; [c] From reference [4]

6.1 References

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7.0 FUTURE RESEARCH & DEVELOPMENT DIRECTIONS

There is always the possibility that a new "silver bullet" chemical formulation will be discovered which turns fine tailings into a material which can easily and economically be handled. It seems that dedication of major resources at this time to such a search is not warranted. Hundreds of chemicals have been surveyed over the last 20 years without generating commercial success. However, one of the contributions from this five year effort has been the development of a protocol which rapidly assesses the benefits of a proposed fine tailings treatment, so that as new formulations are proposed the can be unambiguously evaluated.

The focus of work over the next 1 to 3 years should be on large scale testing of calcium-based treatments of fine tailings. A working hypothesis for the performance modifying behavior of calcium-based treatment has been proposed. While there will continue to be discussion around the specifics, and current knowledge is not exhaustive, it would appear that sufficient knowledge is in hand to guide further large scale testing. There is urgency to bring nonsegregating and freeze-thaw technologies to a decision stage.

The major issues remaining include an assessment of the environmental implications associated with the dewatered tailings deposits. While preliminary analysis does not seem to indicate major concerns, concentration of effort in this area is warranted to establish the necessary level of certainty. Work has been underway for about 6 months to determine the nature of the resulting runoff and seepage water through basic characterization and quantification of acute and chronic toxicity. Generic testing must be augmented with environmental impact assessment within a site specific context. Also important is the ecological performance of the resulting tailings deposits as soil media (see Volume II, Chapter 4.0). Determination must be made as to whether revegetated communities will be self sustaining in the long term reclamation context. Studies are presently underway to determine whether vegetation will take up contaminants and, if so, whether contaminants will be passed into the food chain. It currently appears that the probabilistic risk assessment techniques will be utilized to establish level of ecological risk associated with various reclamation scenarios.

As a final point, one can not resist passing along some recent data and interesting speculation. This is done here as an example of how R&D really works, and why most members of the Consortium get up in the morning. The Consortium sponsored a study of the comparison of OSLO Hot Water Extraction (OHWE) fine tailings properties and behavior to that of Clark Hot Water Extraction (CHWE) through pilot plant testing. When CHWE tailings samples were treated with small amounts of calcium sulphate and placed in 2 meter columns, full consolidation was achieved after 250 days. The most impressive results were provided by samples of Suncor and Syncrude fine tailings that were were treated with 600 ppm of lab grade calcium sulphate, the results of which are shown below.

	Suncor		Syncrude	
	Control	Treated	Control	Treated
Settled volume	0.74	0.56	0.66	.49
Average % solids	29	35	36	39
Degree of consolidation	22	85	31	95

These data show that there has been a significant improvement in properties which may provide a new direction for further investigation. It is often the unplanned discoveries which arise within otherwise structured research work which generate new and exciting directions.

INDEX

VOLUME IV

NON-CLARK EXTRACTION PROCESSES AND THEIR TAILINGS CHARACTERISTICS

VOLUME IV

CONTENTS

1.0 OPPORTUNITIES FOR REDUCTION OF FINE TAILINGS DURING OIL SANDS EXTRACTION

1.1 Objective

This chapter is intended to lend a historical perspective to the development of (and an introduction to the science involved in) five alternatives to the commercial bitumen extraction process. Subsequent chapters provide more detailed descriptions of each technology, their perceived benefits, and the challenges to be met before they can be implemented commercially.

1.2 Introduction

Commercial development of the Athabasca oil sands is widely regarded as one of the most significant engineering achievements in Canada's history. Syncrude Canada Ltd. and Suncor Inc. now operate profitable enterprises in the Athabasca deposit. They produce synthetic crude oil suitable for refining to consumer products from a mixture of sand and an extra-heavy oil once thought to be best suited for paving roads.

The basis for their operations is the Clark Hot Water Extraction (CHWE) process in which mined oil sand is mixed with hot water and caustic (a dispersing agent) to separate the bitumen from the sand. The slurry is further diluted with water, and the bitumen is recovered in a series of flotation cells. Water and solids which float with the bitumen are removed from it and the clean bitumen proceeds to a primary upgrading step.

Each day, Syncrude and Suncor combined, process approximately 465,000 tons of oil sand. Recovery, on average, exceeds 90% of the bitumen in the feed to extraction plants. The 85 million barrels of synthetic crude oil produced each year now correspond to about 18% of Canadian consumption. This proportion will increase as conventional reserves decline and new developments are brought on-stream in the region [1].

Over the years, research scientists and operators have noted that in the caustic-based CHWE process the process chemistry that resulted in the highest recovery of bitumen also coincided with a tailings which, in the short term, had the poorest settling characteristics. With the CHWE process in place, one price for maximizing the utilization of the oil sands resource, is the presence of settling ponds that are much larger than originally anticipated.

These ponds serve two major purposes. They are settling lagoons that allow the extraction plants to operate over the long term with 90% recycled water. They are also storage facilities for the large volumes of fine tailings produced by the CHWE process.

The water surfaces of the tailings ponds at Syncrude and Suncor now cover, in total, almost twenty square kilometers and the mature fine tailings occupy well over 300 million cubic meters [2]. Without further treatment of the existing fine tailings and without process modifications to reduce the rate of production of "new" fine tailings, by the year 2030, over one billion cubic metres of a non-consolidating fine tailings would exist at the bottom of these lakes.

Therefore, while process efficiencies in mining, extraction and upgrading remain to be implemented, the greatest challenge to the oil sands industry lies in the long-term management of the fine tailings. To meet this challenge, a cooperative research program was undertaken jointly by Syncrude, Suncor, OSLO, and various government agencies. This volume describes in detail the experimental work that supported the development of several processes which have a significant potential for reducing the production of fine tailings.

1.3 Alternatives to the Clark Hot Water Extraction Process

Once Suncor and Syncrude had demonstrated that commercial extraction of bitumen from oil sand could be viable, interest in developing alternatives to the CHWE process grew. By the mid-1980s, over 60 alternative extraction processes had been patented [3]. Since then numerous processes have been added to the list.

Although some have shown considerable promise, by early 1995, only Syncrude's field demonstration of pipeline transport of oil sand exceeded a capacity of twenty tonnes per hour of oil sand feed (300 barrels of bitumen product per day). No formal applications for commercial projects had been submitted that took advantage of these alternative processes. The risk of investing large amounts of money in a commercial operation founded on a technology that is unproved in relation to CHWE is a clear impediment to introducing new technology.

For processes not involving water, such as solvent extraction or retorting, successively larger extraction facilities must be built and operated to solve technical issues associated with scale and thereby reduce the risk. Given the high cost of these semi-commercial/demonstration plants, such developments have not occurred.

It would appear that processes that are adaptations to CHWE should have traveled a quicker route to commercial application than "step-out" technologies. Existing plants could be the final testing grounds for modified CHWE processes and perhaps, as well, the first sites for commercial use. Full-scale tests have been carried out in the past at Suncor and Syncrude. However, for several reasons, testing in existing plants may not always provide the anticipated benefits.

First, the limitations imposed on process testing by the design and operational requirements of existing equipment (which has been optimized for CHWE) may obscure the potential benefits of any new process as envisioned by its developer. While the author has no first-hand knowledge of such an occurrence on a commercial scale, experiences with bench-scale hot water process units illustrate the point.

In the late 1970s Syncrude Canada Ltd. designed a cube-shaped, bench-scale batch processing unit that was used originally to estimate the processability of oil sand from Syncrude's lease and its sensitivity to chemical conditions. Subsequently, a unit of cylindrical design was developed at the Esso Research Laboratories for the same purpose.

A direct comparison of the performance of the two units indicated vastly different responses to changes in process variables such as chemical addition and temperature. It was concluded that the geometrical configuration and operation of the units could exaggerate or attenuate the effects of changes in process variables to varying degrees [4].

The description of the initial piloting of the ZEFTE process in Chapter 6.0 (this volume) is another example of a mismatch between the design of existing equipment and the intent of a new process. If this mismatch had not been recognized and understood during evaluation of the first pilot tests, further testing of ZEFTE might not have been justifiable. Within this context, it is easy to envision that the design of existing commercial equipment might not always allow the potentially positive effect of a variable in a "new" process to be seen.

Second, extraction processes are highly complex. Improvements to one stage of CHWE may introduce problems at another stage. For example, introduction of cold water extraction technologies carries with it a new set of challenges associated with cold froth transport. A simple modification such as temperature reduction therefore has the potential for becoming remarkably difficult to implement within the context of the overall extraction circuit.

Third, no demonstration completely removes all risks. One only has to remember that it wasn't until 1965 that the first commercial oil sands operator, Great Canadian Oil Sands Ltd., predicted the accumulation of fine tailings. In Camp's [5] words, "The historical literature on the hot water process for recovery of bitumen from tar sands contains little in the way of hints that a net accumulation of liquid tailings or sludge would occur." The reader should recall that numerous independent pilot tests of extraction had occurred before that time.

Despite the hurdles to implementation of radical innovation in commercial oil sands processing, numerous corporations and government agencies, to their credit, have recognized the wisdom of trying to find improved alternatives to existing technology. Table 1-1 summarizes the state of development of promising alternatives to CHWE that have undergone substantial pilot testing. Additional notes about some of these processes are provided in reference [6].

Table 1-1 suggests that, with the possible exception of ZEFTE, at the time of conception many of these processes originally addressed issues of cost and extraction efficiency. In many cases the processes were subsequently re-engineered to some degree to enhance the character of the tailings. References [7–16] listed in the table are intended to provide the reader with a historical perspective on the development of the processes. More up-to-date references to these same processes, wherein the environmental benefits are described, may be available through Alberta Oil Sands Information Services [17].

1.4 Approaches to Management of Fine Tailings

To place the succeeding chapters of this volume in context it is helpful to understand, in scientific terms, what is meant by "fine tailings." Fine tailings are comprised of mineral particles over 90% of which have an equivalent diameter less than 11 microns [18]. It is hypothesized that very fine particles less than 0.4 microns in diameter are key contributors to the stability of fine tailings. These particles, referred to throughout the volume as "ultra-fines," rapidly form a weak gel structure that retains water after oil sand has been processed by CHWE.

The processes described here have been evaluated in varying degrees of detail by the Fine Tailings Consortium. All have the potential for reducing the volume of fine tailings. Their approaches fall into one of three categories:

- Non-aqueous processes.
- Processes in which the fine tailings formers are managed within the process flow sheet.
- Processes which diminish the potential for fine tailings by removing fine tailings formers from the process in a distinct, engineered process step.

1.5 Five Alternatives to CHWE

1.5.1 AOSTRA-Taciuk Process

Of all of the processes which were developed as alternatives to the CHWE, none had such obvious potential for eliminating the fine tailings problem as the AOSTRA-Taciuk (ATP) retorting process [7]. It was conceived at UMA Engineering Ltd. in 1975 as a means of simultaneously extracting bitumen from oil sand and partially upgrading it.

TABLE 1-1. A summary of alternatives to the CHWE Process

Process name	Developer	Date of invention	Scale of testing	Principle of operations	Original incentives for development	Effect on tailings
AOSTRA-Taciuk Process [7]	UMATAC Industrial Processes; Alberta Oil Sands Technology and Research Authority	1974	5 tph	Retorting	• Reduced costs to produce a partially upgraded, pipelineable oil. • Reduced effect of grade on recovery. • Simplicity of design and operations. • Energy self-sufficiency. • Production of dry tailings.	• Good settling properties of solid tailings even if slurried to disposal site.
BITMIN [8]	Kilborn Eng. Ltd.	1990	20 tph	Water washing	• Simplicity of design and operation. • Reduced capital costs. • Reduced energy requirements.	• Production of a geotechnically stable tailings.
CanOxy Sand Reduction [9]	Canadian Occidental Ltd.	1991	2 tph	Water washing	• Rejection of sand at mine site leading to reduced material handling costs.	• Reduced size of tailings pond.
Dow-Kellogg Process [10]	Dow Chemicals Co.; Kellogg Rust Synfuels Inc.	1980	Pilot	Solvent extraction	• Increased recovery from low grade ore. • Reduced effects of clays on recovery. • Reduced sensitivity to ore oxidation. • Use of off-the-shelf equipment. • Low water consumption.	• No tailings pond.
Dravo Process [11]	Dravo Engineers Ltd.	1980	8 tph	Solvent extraction	• Increased recovery from low-grade ore • Suitable for oil-wetted sands	• Production of damp tailings suitable for immediate back-filling of mines
Gulf/Solid RTR [12]	Rio Tinto TIL Holding S.A.; Gulf Canada Ltd.	~1975	20 tph	Water washing	• Energy savings due to internal recycle of water • Process development for "modular" isolated plants • Reduction of clay dispersion during processing leading to increased recovery from low grade ores	• Production of a solid tailings
OSLO Cold Water Extraction [13]	Esso Resources Canada Ltd.	1986	20 tph	Water washing	• Extraction process to be integrated with wet mining techniques	• Enhanced settling and strength characteristics of fine tailings
OSLO Hot Water Extraction	Esso Resources Canada Ltd.	1986	Commercial demonstration tests	Water washing	• Retrofitting of existing commercial operations to take advantage of enhanced tailings properties observed in OCWE process	• As above
Oleophilic Sieve [14]	Oleophilic Sieve Development of Canada Ltd.	1975	20 tph	Water washing	• Improvement in bitumen recovery by replacing oil flotation with capture by agglomeration and adhesion of oil to an oleophilic sieve	• Reduced oil losses to fine tailings; process can be used to recover oil from mature fine tailings
SESA [15] Damp	National Research Council	1965	0.6 tpd	Solvent extraction/spherical agglomeration separation in a water medium	• Overcome solids/liquid separation problem in solvent extraction processes • Increased recovery of bitumen from low grade ores • Low water make-up • Elimination of chemical process aids	• Production of a solid tailings
Solv-Ex [16] Damp	Solv-Ex Corporation	1991	2 tph	Solvent assisted water extraction	• Processing of oil-wet sands • Improved recoveries from low-grade ores • Reduced capital and operating costs • Reduced operating temperature	• Production of a tailings
Zero Fine Tailings Extraction	Syncrude Canada Ltd.	1992	2.5 tph	Water washing	• Reduction of fine tailings volumes from existing operations	• Production of "engineered" tails within the extraction circuit

In the twenty years following conception of the idea numerous laboratory, pilot, and engineering design studies have been completed to improve upon the technical features and economics of the process. The technology has advanced to the stage where a 10-tonne per hour processor is in commercial use for cleaning up oily wastes. A significant effort was recently completed by industry in cooperation with UMATAC to overcome perceived impediments of the ATP as they related to oil sands extraction. The reader is encouraged to contact the Oil Sands and Research Division of the Alberta Department of Energy for this information [20].

Were it not for the need to improve process economics wherever possible, there would be little reason to discuss the ATP in this volume. No process which involves retorting at 800°C and adding water to the spent sand solely for the purpose of quenching it can realistically be viewed as a process that has potential for producing fine tailings.

However, economics did come into play. The original concept for a commercial process involved conveying or trucking the quenched sand back to the mined-out areas. More recent experience suggested that in fact hydraulic transport of the spent sand could reduce disposal costs.

Would this approach recreate the fine tailings problem which had been circumvented by the processing end of the technology? Chapter 8.0 provides a detailed description of the testing done to examine the settling properties of the spent solids from the ATP retort after they are dispersed in water.

1.5.2 Zero Fine Tailings Extraction (ZEFTE)

The second category of processes to reduce production of fine tailings is that in which the fine tailings formers are managed within the process. The Zero Fine Tailings Extraction (ZEFTE) process clearly embodies that philosophy.

Early beliefs about the formation and stability of mature fine tailings tended to be centered around the concepts of either hindered Stokes' settling or a net repulsive force that existed between individual fine (or ultra-fine) particles. Undoubtedly those beliefs grew from the observation that the conditions for maximum bitumen recovery and highest froth quality coincided with a high degree of fines dispersion during processing and a relatively slow initial settling rate. Under these circumstances could fine tailings be anything but dispersed fine particles undergoing hindered settling?

Only after a detailed investigation of mature fine tailings themselves was it determined that they are in fact a collection of flocs with a weak gel structure that retains water under the conditions of the tailings pond [19]. The lower limit for water content of mature fine tailings appears to be in the neighborhood of 70%.

ZEFTE was conceived at Syncrude Canada Research Ltd. in 1992, as a result of considering simultaneously some basic engineering principles and the observations made previously. It was based on the hypothesis that if recovery was affected favorably by keeping fines dispersed during extraction, and if mature fine tailings resulted from some sort of gel or floc formation, then the creation of a chemical environment that would keep the fines permanently dispersed could benefit both extraction and tailings management.

Within this context, water with a relatively high level of dispersed fines could be recycled immediately within the process before froth quality and recovery would be affected. The basic engineering principles that bring this concept to life are amazingly simple.

The standard separation vessels at Syncrude and Suncor have inlets approximately at their midpoints. Given that there is withdrawal from these vessels from both the top (froth overflow) and bottom (sand underflow), a cut point defined as the size of particle which will not report to the froth, can be calculated for the net vertical velocity of the fluids within the separation vessel. With an appropriately designed vessel, operated under well-defined conditions, the cut point can be controlled.

On theoretical principles then, during start-up solids below the cut point (including the ultra-fines) would build up in the recycle water. However, at some point the introduction into the extraction circuit of ultra-fines present in the oil sand feed would be balanced by the loss of the same species to the sand underflow stream. In effect, the sand underflow from the separation cell would be an "engineered" tailings stream. Fresh water would be required only to make up for the volume of water in those engineered tailings.

Chapter 6.0 describes the laboratory and pilot programs that were conducted to determine if this concept could be viable on a commercial scale. These programs met all expectations concerning bitumen recovery even though the solids content of the recycle water was ten times greater than what was previously viewed as being the upper limit in CHWE. Measurement and understanding of the implications of the geotechnical properties of beached, engineered tailings will be the next major step in the development of the process.

1.5.3 BITMIN

The BITMIN process was developed at Kilborn Engineering Ltd. in 1990 for the purpose of improving the extraction circuit. It has since evolved to the point where it yields substantial environmental benefits.

The Strand separator, the key vessel within the BITMIN process, was designed as a solids rejection unit serving the same general purpose as the tumbler and the primary separation vessel in the CHWE process. Even at the time of conception, the possibility of reduced clay dispersion as a result of gentle conditioning without the use of caustic, was acknowledged.

Over a period of time, given the realities of water management, a flow sheet was developed that minimized consumption of fresh water. The approach involves removing fines from the water prior to recycling it through the extraction circuit. This is accomplished by treating waste water streams with proprietary polymers at a controlled level of bicarbonate to flocculate the fines.

The flocculated fines stream is then mixed with the coarse solids from the Strand separator. The mixture is dewatered on a vacuum filter belt to produce an engineered tailings with suitable geotechnical properties for backfilling the original oil sands mine. It is intended that backfilling will follow mining by three years with re-vegetation of the tailings dump two years thereafter.

Chapter 7.0 of this volume provides considerably more detail about the BITMIN process with particular emphasis on the geotechnical and environmental properties of the engineered tailings. To date, the performance of the process has met or exceeded all expectations. Integration of an engineered tailings disposal system into the process was a late addition. In the near term, the next area of focus will be an understanding of the properties of these tailings and an assessment of the potential to reclaim and re-vegetate the landscape within five years of mining.

1.5.4 OSLO Cold and Hot Water Extraction Processes (OCWE and OHWE)

The OCWE process [12] was developed in the late 1980s by Esso Resources Canada Ltd. in an attempt to realize the efficiencies of dredging and pipeline transport of slurried oil sand to a bitumen extraction plant. A conditioning agent, comprised of a flotation agent such as kerosene or diesel and a frother such as methyl-isobutyl-carbinol, when added at the front end of the transport line would improve the recovery of the bitumen when the slurry reached the separation vessel in the extraction plant.

Very quickly the favorable properties of tailings from the OCWE process *vis-à-vis* the CHWE process were noted, and the OHWE process was conceived to permit existing CHWE-designed plants to produce a fine tailings stream with the same characteristics as OCWE tailings.

A significant amount of work has been undertaken to understand the reason for the difference in behavior between the OCWE/OHWE fine tailings and CHWE fine tailings. These studies are the subject of Chapter 3.0.

The OCWE and OHWE processes are ones in which the fine tailings formers are managed in terms of their chemical properties and removed by natural settling processes. Their non-caustic chemical environment reduces the extent to which the fine tailings formers are dispersed in the water. Particle size distribution and surface area are obvious indicators of this phenomenon.

A second advantage of the OHWE and OCWE processes relates to the structure of the fine tailings as they mature. Fine solids generated by the non-caustic processes not only settle more rapidly because of their larger size but orient themselves in a manner that is conducive to self-weight consolidation at a much lower solids content. Detailed comparisons of geotechnical properties of CHWE and OWHE/OCWE fine tailings also follow in this volume.

Given that the OHWE process is intended to be used in the existing designs of commercial plants, there has been an opportunity to carry out head-to-head comparisons of the OHWE and CHWE processes. Testing at a scale of 2.5 tonnes per hour using oil sands of just over 12% bitumen has indicated that recoveries with the OHWE process are in the neighborhood of 1% lower than those from the CHWE process. A 10% reduction in primary recovery of bitumen in OHWE is compensated for the most part by subsequent scavenging stages. The bitumen content of the combined OHWE froths average about 2% less than that of the combined CHWE froths [23]. No statistical significance has been attributed to these apparent trends.

These similarities in performance indicate that any losses (if they in fact exist) incurred in production by converting to OHWE may be more than offset by reduced costs in long-term management of tailings. Given the objective of converting existing operations to the OHWE process without re-design of the plant, the proponents of the process are concentrating on gaining an understanding of the tailings properties rather than on improvements to the extraction circuit.

One issue which remains to be addressed is the implication of implementing the OHWE process in existing plants which rely on extensive recycle of water from tailings ponds generated from the CHWE process.

1.6 Summary

Existing and potential operators in Alberta's oil sands have accepted the challenge to reduce the environmental impact of the current commercial operations and to ensure that fine tailings are not a subject associated with future oil sands plants. In this chapter, you have been introduced to the free-thinking and innovation that may meet these objectives.

How will the developers of new extraction processes prove their value to an investor's satisfaction? The AOSTRA-Taciuk Process has the advantage of generating cash flow (and confidence) through its commercial application in environmental clean-up and perhaps in the area of oil shale retorting.

Immediate opportunities for demonstration of the other processes described here are not so obvious. However, the apparent trend to commission satellite bitumen production operations may be the opportunity for these technologies to succeed at a lower level of risk than was previously possible. Until then, a well-planned approach to technology development that integrates demonstration on suitably designed equipment, experience from existing commercial operations, and an understanding of the science of the processes is the only apparent path to follow.

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2.0 STATUS OF OCWE PROCESS AND TAILINGS MANAGEMENT RESEARCH AND DEVELOPMENT

2.1 Abstract

During the 1986 to 1990 period the OSLO joint venture, a consortium of six companies (Alberta Oil Sands Equity, Canadian Occidental Petroleum Ltd., Imperial Oil Resources Limited (the operator), Gulf Canada Resources Limited, PanCanadian Petroleum Limited, and Petro-Canada Inc.), and Alberta Oil Sands Technology and Research Authority (AOSTRA) developed two bitumen extraction processes for the Athabasca oil sands, namely the OSLO Cold Water Extraction and the OSLO Hot Water Extraction (OCWE and OHWE) processes. Both processes use methyl-isobutyl-carbinol (MIBC) and kerosene to enhance bitumen flotation, but the operable temperature ranges are 5 to 30°C for the OCWE process and 30 to 90°C for the OHWE process.

Each process produces fine tailings that settle fast and gain enough strength to support a layer of coarse sand. These characteristics offer advantages over caustic-based processes in certain aspects of tailings management and lead to unique concepts such as the inter-layering of coarse and fine tails to further consolidate fine tailings thus creating reclaimable solid tailings.

From 1986 to 1990, process and tailings development work for the OCWE process proceeded from bench-scale batch testing to 2-tph laboratory pilot testing and from there to 20-tph field pilot testing. Both the OCWE and the OHWE process are considered ready for large-scale demonstration tests and commercial exploitation.

2.2 Introduction

In 1986 OSLO joint venture and AOSTRA (now Alberta Department of Energy — Oil Sands and Research Division) began investigations to develop low cost bitumen extraction processes and invented two processes, namely the OCWE and the OHWE processes. From 1986 to 1991 most of the research and development work was aimed at developing the OCWE process and understanding its tailings properties. Limited testing of the OHWE process indicated that fine tailings from both processes possessed similar characteristics.

During 1993 and 1994 the Fine Tails Fundamentals Consortium carried out a comprehensive study to compare the properties of fine tailings generated by the OHWE and the Clark Hot Water Extraction (CHWE) processes, and the results are reported in Chapter 3.0 of this volume.

This paper reviews the research and development work carried out to-date on OCWE process tailings. It is noted that most of the research and development work on this process was carried out from 1986 to 1991 and that no further research has been done since then. An 800 to 1200-tph demonstration pilot project, that would have been located either on OSLO Lease 31 or at the Suncor Mine, was planned but never installed. Limited research and development work on tailings continued until 1994.

Both OSLO processes offer a reclaimable tailings disposal scheme as an alternative to the wet landscape disposal scheme being proposed for fine tailings from the CHWE process. The economic advantage and the environment impact of the solid tails management system over the fluid tails management system needs to be determined on a project-by-project basis.

The next step in the development of the OSLO processes and schemes for managing their respective tailings is a large-scale (1000+ tph) demonstration. This program should evaluate several tailings disposal schemes such as the inter-layering of fines and sand, enhanced fines consolidation using freeze-thaw cycling, and modified beaching and fluid tails containment.

2.3 The OCWE Process

Figure 2-1 is a schematic process flow diagram. The OCWE process was invented to extract bitumen from dredge-mined oil sand where the oil sand is slurried at the submerged, dredge cutting head and pumped through a pipeline to flotation vessels in the process plant. Dry-mined oil sands could also be slurried near the mine face and pumped to the extraction plant (Figure 2-2). Two chemicals (MIBC and kerosene) are used to condition the slurry in the pipeline en route to the extraction plant, and air is introduced into the pipeline to generate air bubbles for bitumen flotation. Bitumen attaches itself to the bubbles and is floated to the surface in the flotation vessels where it forms a bituminous froth. Two-stage flotation vessels were employed in the 20-tph field pilot on Lease 41 during which the operating temperature varied between 5 and 30°C.

Tailings from the separation vessels are pumped to the tailings pond where coarse sand settles to form dykes and beaches and fines, carried by the runoff water, accumulate in the middle of the pond. The solids concentration in beach runoff varies between 7 and 15 percent by weight. Dewatering of the fines occurs in the tailings pond and, in about six weeks, the fines have settled to a solids concentration of 25 to 30 percent by weight. The clear water layer above the fines is recycled to the process. There are several ways of consolidating the partially dewatered solids to a dry solid state. This paper reviews the results of the concepts tested.

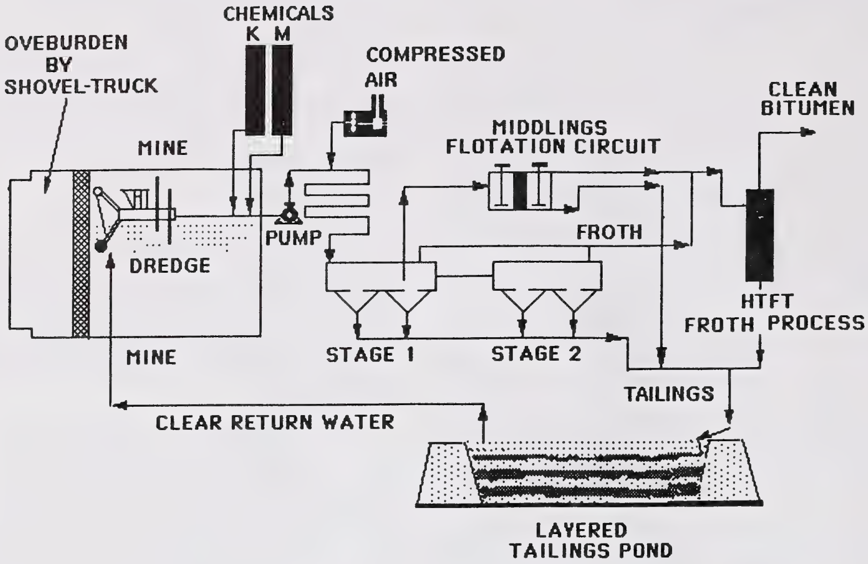

FIGURE 2-1. Conceptual OCWE process flow sheet.

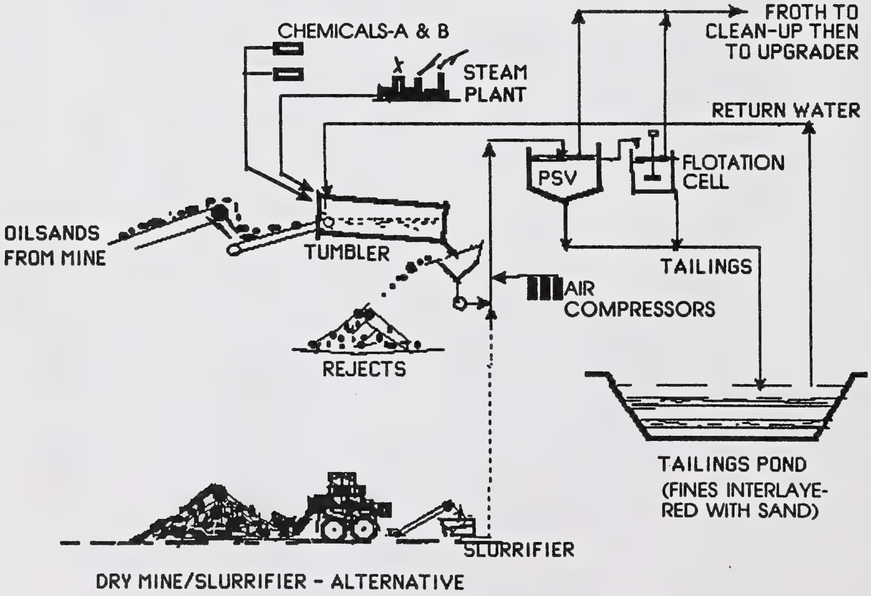

FIGURE 2-2. Dry mine/OHWE process schematic layout.

2.4 Laboratory Tests on OCWE Process Tailings

It was realized during bench-scale batch testing of the OCWE process in 1986 and 1987 that fine tailings settled rapidly and that clear water was released for re-use within one to two days. The same phenomenon was observed during subsequent 2-tph pilot-scale testing. In this program, tailings tests included small (100 mm diameter) cylinders and fish tanks and the fines settled quickly and consolidated to a mature state within a few days. They were then layered with coarse sand by pluviating sand through water. These cursory tests indicated that fine tailings could be solidified by inter-layering settled fines and sand.

OSLO's in-house studies suggested that managing solid fine tails could offer an economic advantage over the industry's present system of managing fine tailings in a fluid state. This led to the initial tailings research (1986–1988) work that was carried out at Komex, CANMET, and Golder Associates laboratories and which included:

- Particle Size Distribution (PSD) of fine tailings by the standard ASTM method.
- Layering of fines with coarse sand.
- Determination of the structure of settled OCWE fine tailings using an Electron microscope.
- Comparison with CHWE fine tailings (from the present commercial mine tailings pond).

The following conclusions were drawn from this work:

- The PSD of OCWE fine tailings is similar to that of CHWE fine tailings.
- At 30 weight percent solids, the strength of settled fines (Figure 2-3) was two to three times higher than the strength of CHWE fines at similar solids concentrations.
- The higher inter-particle strength led to the application of sand layering for consolidation of OCWE fines. A limited comparative layering test on CHWE fines resulted in failure.

2.5 Lease 41 Pilot: Tests on OCWE Process Tailings

To fully develop the OCWE process [1], field pilot tests were conducted on OSLO's Lease 41 from 1988 to 1990. The Lease 41 test site (Photo 2-1) was located about 10 km north of Fort McMurray just off highway 63. Oil sands for the tests were obtained from the Suncor mine and placed in a trench about 5 m wide × 3 m deep × 50 m long. A bucket wheel and a pump were mounted on a backhoe boom to mine and slurry the oil sands which were then pumped to extraction flotation vessels (Photo 2-2) *via* a pipeline 76 mm in diameter and about 600 m long (Figure 2-4).

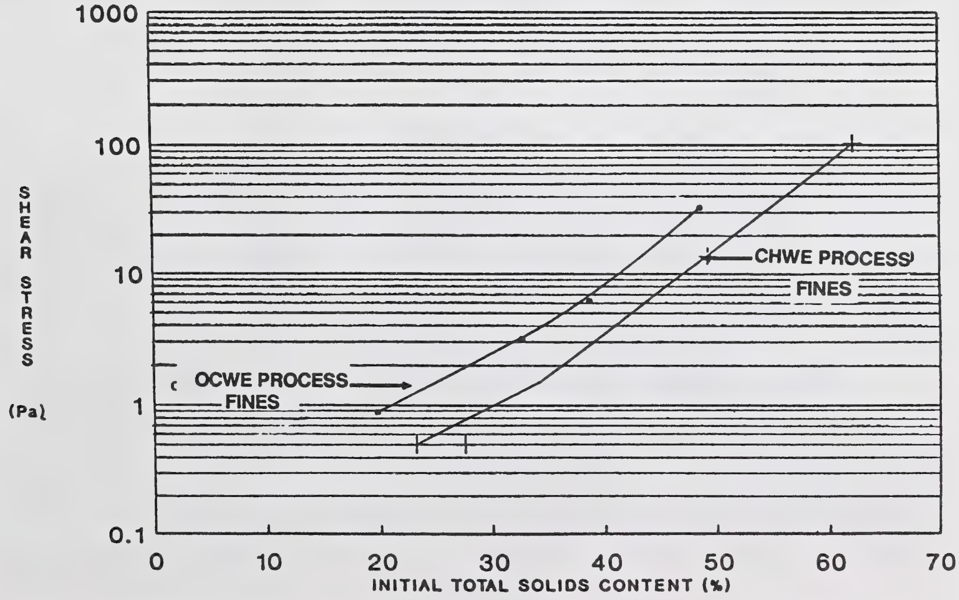

FIGURE 2-3. Solids content effect on shear stress.

PHOTO 2-1. Lease 41 pilot test site.

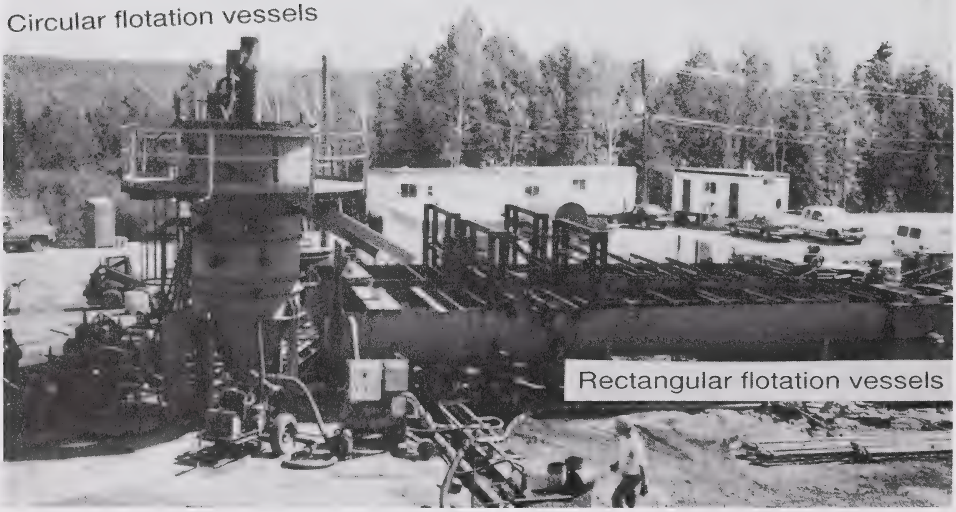

PHOTO 2-2. OCWE process plant at Lease 41.

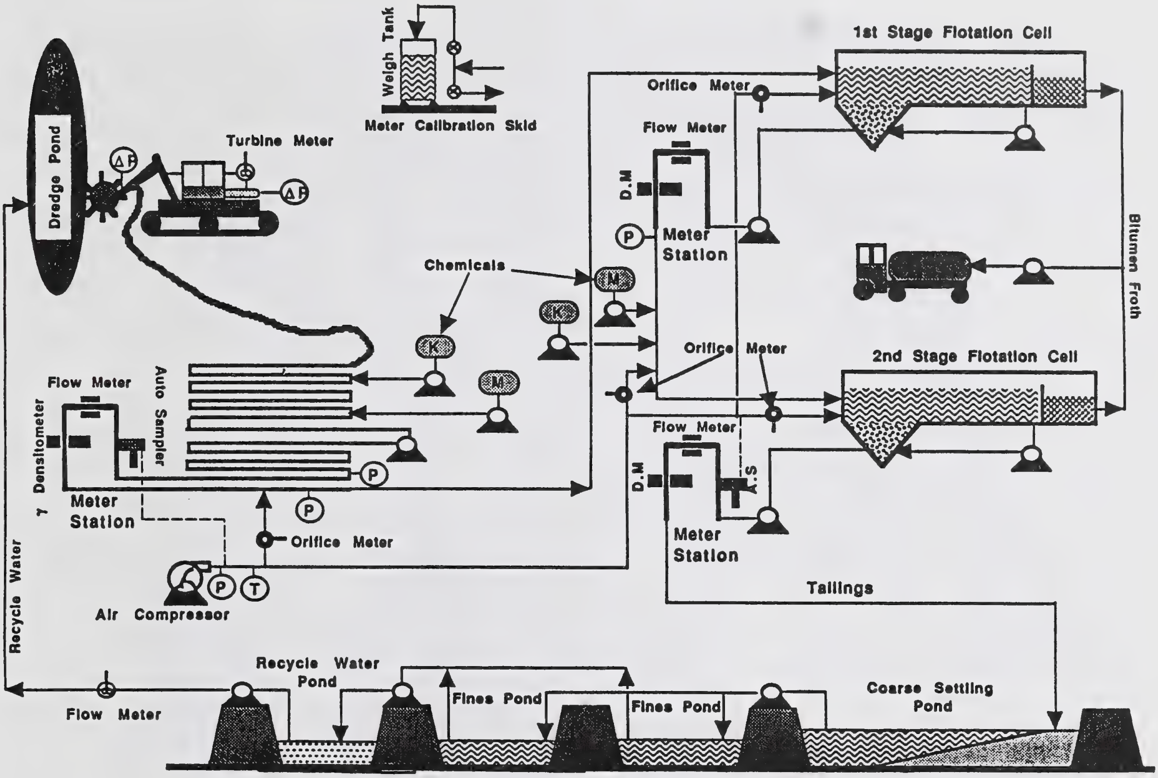

FIGURE 2-4. OCWE process field pilot at Lease 41. 1989 test program conceptual layout.

2.5.1 Tailings Management

As shown in Photo 2-1 and Figure 2-4, tailings from the process vessels were pumped to the "Total Tailings Pond." There coarse sand settled to form a beach and fine solids and water accumulated at the far end of the pond. From the Total Tailings Pond, fine tails were pumped to either Fines Pond #1 or #2 and allowed to settle for 24 to 48 hours to release clear water for re-use in the process. Because the tests were continuous for three to four months, fines never had a chance to consolidate to permit a layering test on an on-going basis.

Hence, the tailings tests were initiated at the end of 1990 following the completion of the extraction tests. At this time, the accumulated fines layer in each of the Fines Ponds was about one metre thick and had a solids content of 15 to 20 percent by weight. The dimensions of the ponds were sufficient to minimize edge effects on sedimentation and consolidation. Each pond was 15 m wide × 3 m deep × 60 – 75 m long.

The following laboratory programs were undertaken in conjunction with the field study:

- Engineering properties of OCWE process fine tailings — Golder Associates Ltd. (Calgary).
- Fines (30% solids) structure by electron microscopy — CANMET (Devon).
- Enhanced consolidation of fines by freeze/thaw method — University of Alberta (Edmonton).
- Design of sand layering and development of equipment for sampling fines — Northwest Hydraulic Labs (Edmonton).

2.5.2 Engineering Properties of OCWE Fine Tailings

The field program was aimed at answering several questions that were key for successful layering of OCWE fine tailings with sand:

- What is the optimum solids content of settled fines needed to support a sand layer?
- How long will it take deposited fines to reach this solids content?

- Are there any limitations regarding the placement of sand on top of fines?
- At what rate will fines consolidate following the addition of sand layers?
- Do the sand layers provide adequate drainage for water released from the fines?
- Can surface freeze/thaw or addition of cement be used to aid sand layering?

2.5.2.1 *Test Work*

Fine tailings for the test work were obtained from Ponds #1 and #2 on Lease 41. They were still in a fluid state but represented aged material because they were accumulated over a three-year period. Fresh fines were obtained from the OHWE pilot tests carried out in 1990. Sand for layering experiments was obtained from Lease 41 beaches.

The test work included the following:

- Soil index tests on all material
- PSD on all samples
- Water chemistry
- Clay/sands mineralogy
- 76 mm and 203 mm diameter glass cylinder tests for sedimentation and consolidation
- 30 cm × 30 cm × 60 cm long fish tanks
- 75 cm diameter × 2.6 m high tanks for 0.3 m to 1 m thick fines layering tests
- Determination of pore pressure of fines
- Vane shear and odeometer strength tests
- Freeze/thaw tank tests on fines

Test results indicated that the fines settle quickly (sedimentation 1 to 2 days), consolidate (hindered settling) to about 30 weight percent solids in a couple of weeks, and possess enough strength (at 30 percent solids) to support a layer of coarse sand.

2.5.2.2 *Conclusions and Discussion*

Following are specific conclusions and corresponding discussion:

- The volume of mature fine tails (MFT) generated by the OCWE process was predicted to be similar to the volume generated by the CHWE process based on PSD data (Figure 2-5) which showed that the proportion of $<2\ \mu m$ particles in settled fines is about 40 weight percent for each process. The 1994 comparison of the OHWE and CHWE processes confirmed these data for fines volumes. However, the amount $<2\ \mu m$ solids in OHWE process fines was actually only 10 percent by weight. Subsequent investigation found that the ASTM procedure explains the discrepancy. This procedure disperses fine solids with either Calgonite (sodium hexametaphosphate) or distilled water both of which act as dispersants. Fines aggregates in OCWE process tailings were thus dispersed and made to behave like the caustic-dispersed fines in CHWE tailings. In essence, the standard ASTM procedure is the wrong procedure for determining the PSD of fines from various oil sand extraction processes and researchers are advised not to use it. The double hydrometer technique should be used instead.
- Layering tests indicate that the solids content of settled fines needs to be about 30 percent by weight before layers of sand can be placed on top of them, and the sand needs to be pluviated through water. It appears that effective stresses first occur in settled OCWE fines at about 30 percent solids. The sheer stress at 30 percent solids is about 2 Pa which in geotechnical terms is considered very low (Figure 2-3).
- Migration of sand through settled fines was minimal at solids contents higher than 30 percent by weight.
- Tests in columns and tanks indicated that it takes only about one to two days for initial settling of fines. During this time the solids content increased from an initial six to eight weight percent to about 15 percent (pre hindered settling stage — Figure 2-6) and about 30 days was required to reach 30 percent solids (hindered settling stage). Table 2-1 shows settling rate projections of various thickness of fines. By way of example, hindered settling takes 30 days for a 0.15 m thick fines layer, 130 days for 0.5 m thick fines layer, and may take 50 to 100 years for a layer 10 to 15 m thick.
- After a sand layer is placed on top of the fines consolidation proceeds rapidly. The thickness of the sand layer is an important factor in determining the time required for consolidation.
- Freeze/thaw tests on fine tails indicated that the method has significant potential for enhancing consolidation of settled fines. The initial solids content of 30 weight percent increased to 45 percent in a single freeze/thaw cycle in both the laboratory and the field tests.
- The Electron Microscopy study on the structure of OCWE fines with 10 weight percent solids showed that they have a different structure than CHWE fines (from Syncrude's tailings pond). This observation was reconfirmed during the 1994 comparison of the OHWE and CHWE processes. OHWE process fines form a random (edge-to-face) structure at 30 weight percent

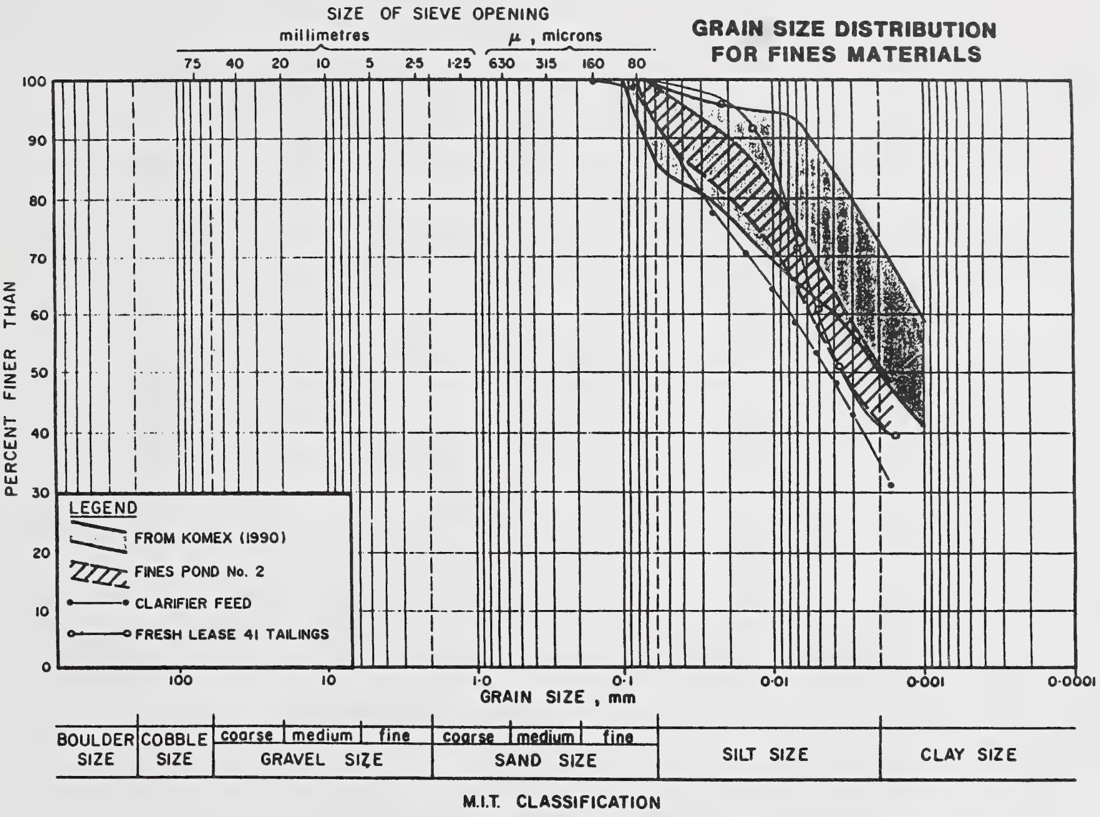

FIGURE 2-5. OCWE size grain size distribution.

solids while CHWE fines form an ordered (face-to-face) structure. The random structure is considered to be stronger than the ordered structure.

- An empirical consolidation model was developed from relationships between effective stress (Figure 2-7) and void ratio and between permeability (Figure 2-8) and void ratio. The former relationship determines the amount of consolidation or compression that *will* take place, while permeability determines the ratio at which consolidation *can* take place. The consolidation equation couples the two relationships. Using this model, it was determined that
  - after 31 days a one metre thick layer of fines settles to a height of 0.89 m with a solids concentration of about 30.3 percent by weight, and that
  - addition of 9 kPa (a one metre thick sand layer) above the fines layer consolidates it to a height of 0.48 m with an average solids concentration of 50 weight percent after 365 days.
- Additional layering tests were carried out at Komex Laboratory [4] in Calgary using a large glass tank (2 m × 2 m × 2 m) with appropriate instrumentation. A fines layer of about a meter thick was place in the tank and loaded with coarse sand. A distinct layer of sand (0.3 m thick) stayed intact even after placing "heavy weights" on the sand layer (Photo 2-3). This experiment indicates that the sand layer provided drainage path and no penetration of fines into sand was observed.

2.6 Lease 41 Pilot: Fine Tails Layering Tests

The primary objective of the field layering test program [2] was to confirm the results of layering tests carried out in various laboratories. A secondary objective was to understand the scale-up relationship between laboratory tests and large-scale continuous tests.

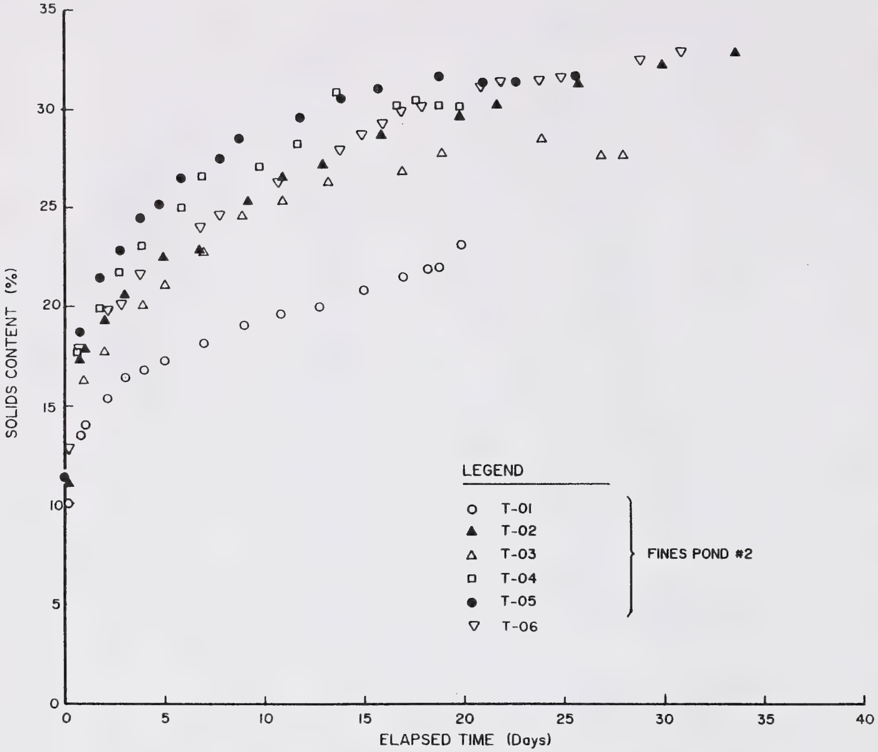

FIGURE 2-6. Solids content *versus* time tank tests.

TABLE 2-1. Settling and consolidation for different layer thickness.

Layer thickness at 30% solids (m)	Sedimentation		Hindered settling		Time to reach 90% consolidation (days)
	Initial height (m)	Time (days)	Initial height (m)	Time (days)	
0.01	0.03	1	0.016	1	1
0.1	0.3	1	0.16	1	6
0.25	0.75	2	0.024	43	12
0.5	1.5	2	0.84	130	48
1	3	2	1.67	928	380
10	30	15	16.7	88 years	>100 years

Notes: 1. Sedimentation from 10% to 18% solids. 2. Hindered settling from 18% to 30% solids. 3. Consolidation above 30% solids. 4. Double drainage conditions assumed. 5. Hindered settling model is not valid for Initial Height. >1m.

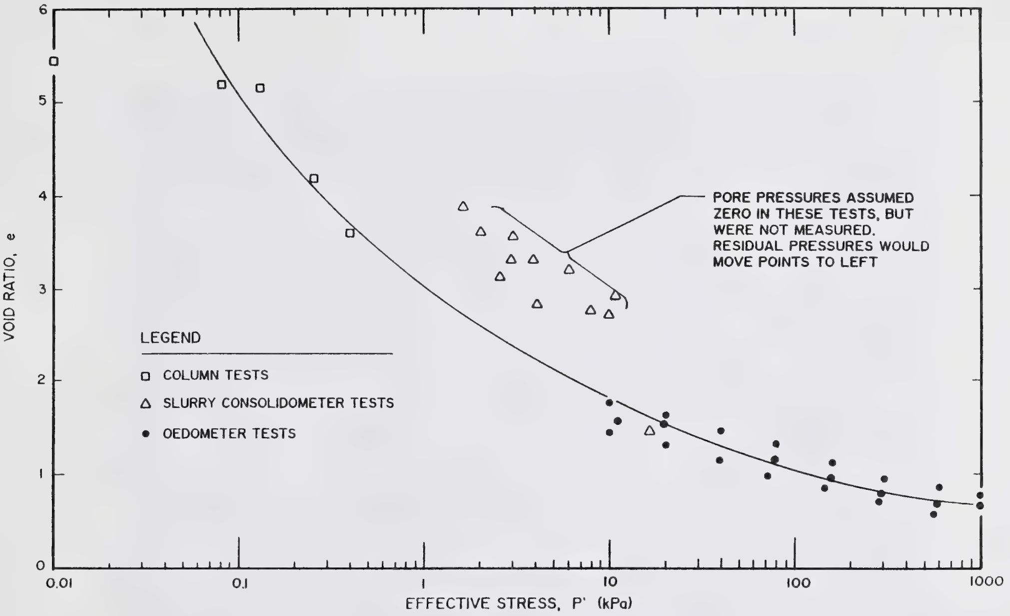

FIGURE 2-7. Void ratio *versus* log effective stress.

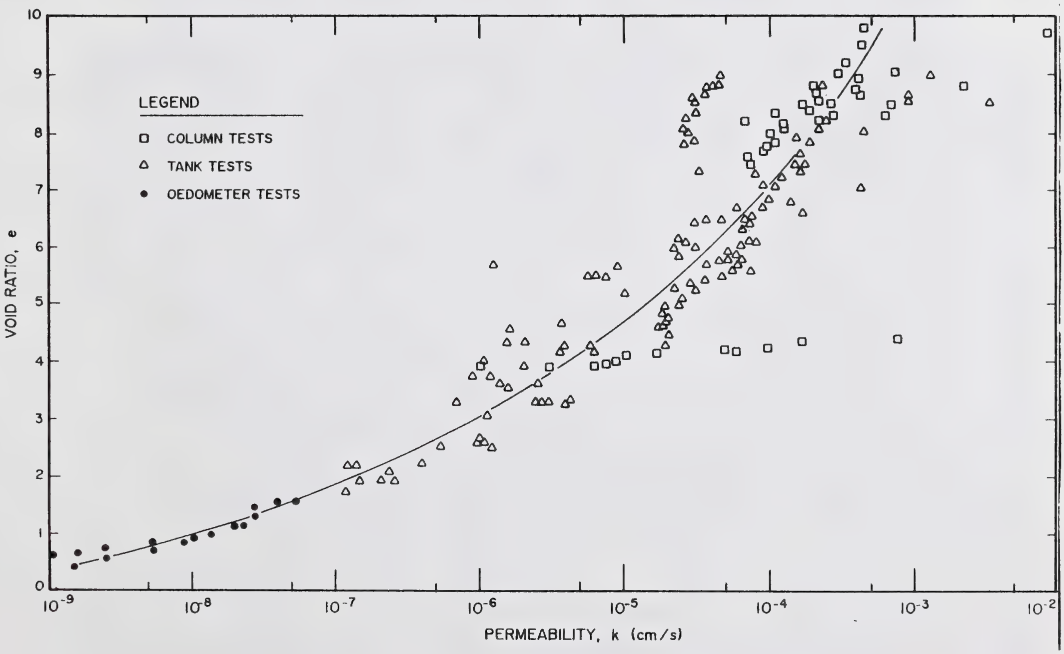

FIGURE 2-8. Void ratio *versus* log permeability.

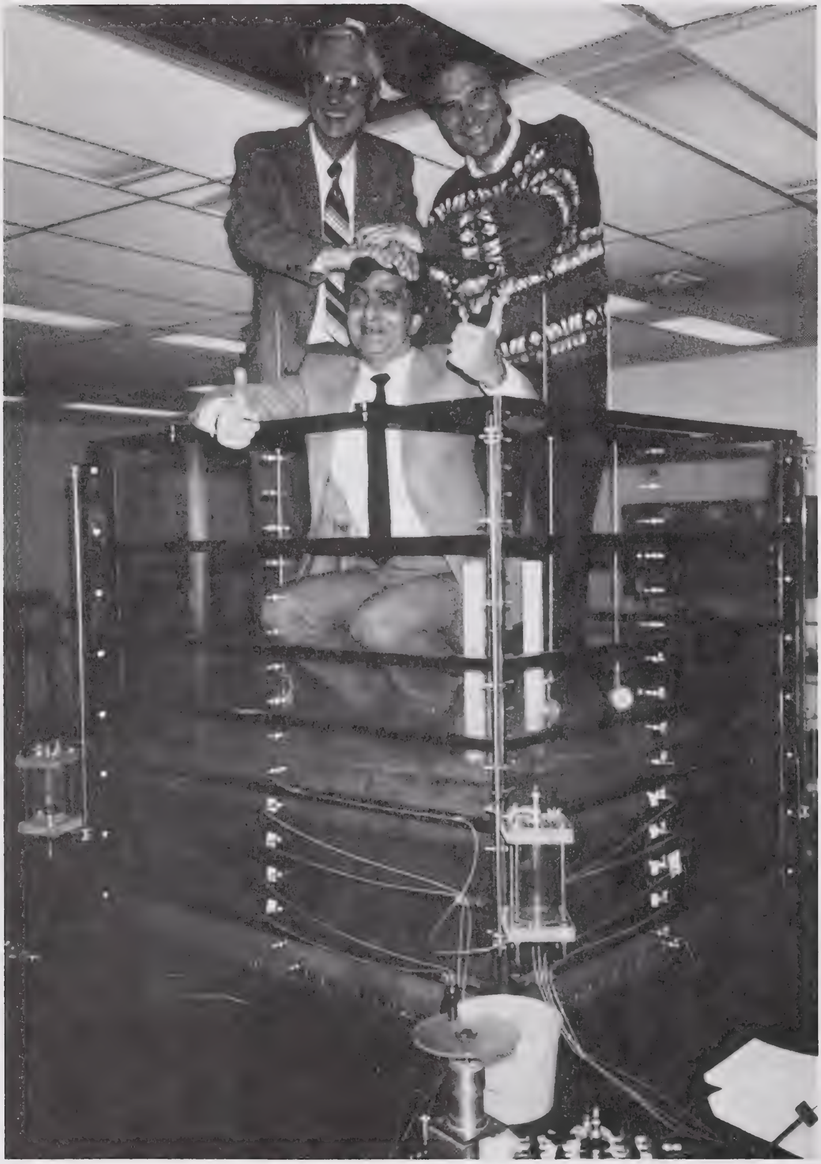

PHOTO 2-3. Fine tails layered over with sand (note heavy weights on the sand layer).

2.6.1 Design of Sand Layering Equipment

Extensive tests were carried out at Northwest Hydraulics Labs in Edmonton to design a simple sand layering buggy that would float on a one-meter thick water layer and spray sand gently onto the settled fines without disturbing them.

A tank 3 m × 1.8 m × 7.3 m long with one glass side was used to test various techniques for spraying sand onto a layer of fines (30 percent solids) that was placed in the bottom of the tank under a 1.0 to 1.5 m layer of water. Sand obtained from the Lease 41 pilot was slurried in a vessel and pumped to the test tank. Several methods of discharging the sand under the surface of the water destabilized the fines layer and a sand layer could not be formed. The acceptable method was one that impinged the sand slurry onto a spoon above the water and spread the sand slurry over the water in a fan-shaped jet. The sand then dropped through water and formed a layer over top of the fines layer.

This design was incorporated into a buggy (Photo 2-4) to layer sand on Lease 41 Fines Ponds #1 and #2. The buggy was also equipped to take measurements of fines at various depths in the fines layer.

2.6.2 Fine Tailings Layering Tests

The engineering data and sand spraying equipment from laboratory test work [3] was used to layer sand on both Ponds #1 and #2 fines.

Fines Pond #1 was 15 m wide × 75 m long and Pond #2 was 15 m wide × 60 m long. Both ponds had a fines layer about 1 m thick that had settled to a density of about 25 to 30 weight percent solids. The spray buggy, as shown in Photo 2-4, was used to distribute sand over the fines so that it was pluviated through a one meter thick layer of water. Coarse sand from the Total Tailings pond was slurried with water jets and pumped to the sand spraying buggy. The initial sand layer sprayed on top of the fines was only a few centimeters thick, but after a week successive layers of sand were added — over a period of months — on both ponds . In total, a one metre thick layer of sand was placed over the fines in Pond #2. However, in Fines Pond #1, when the sand layer reached about 0.35 m about 30 percent of the sand layer toppled over indicating that the strength of the fines was not at its optimum. This is consistent with the laboratory observations that effective stresses start to appear at about 30 percent solids content. The toppled sand area was once again layered over

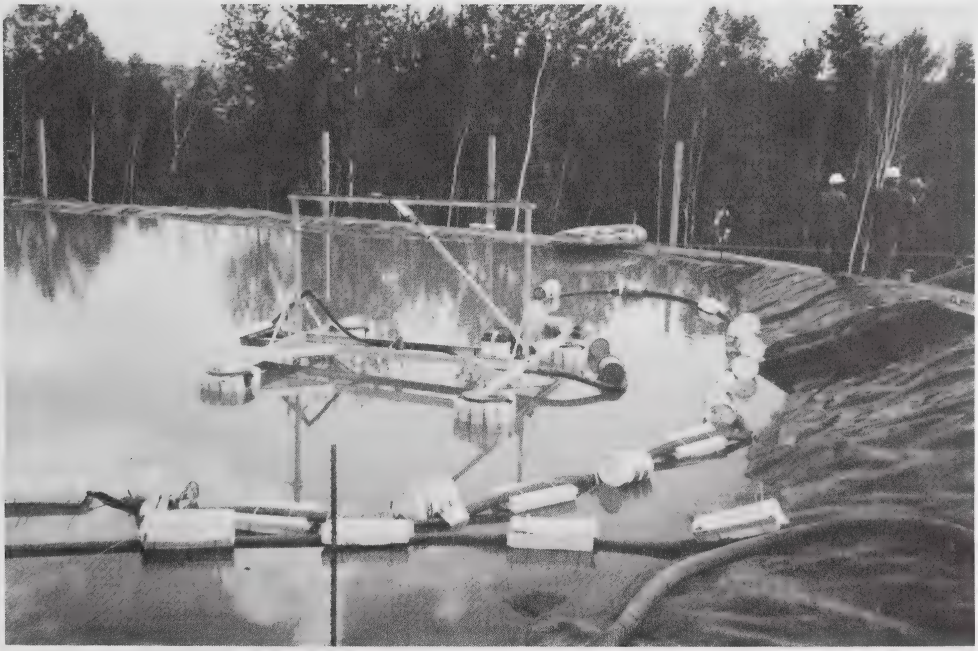

PHOTO 2-4. Sand spraying buggy in Pond #2.

with coarse sand and no failure was observed. Further, fines from the rest of the pond did not penetrate through the toppled area indicating that the layering process can be self-healing.

Appropriate samples were taken regularly at three locations along the long centre line of each pond (one in the centre and one near each end) over the 1991–92 period. The one meter thick layers of fines that started with about 30 percent solids in 1991 had consolidated to about 0.5 m and the solid contents had increased to about 70 weight percent by the end of 1992 — almost to a natural soil state (Table 2-2). During 1993, both ponds sites were layered over with muskeg and top soil and the area was seeded. The area is now reclaimed.

2.6.3 Freeze-Thaw Tests

After the sand layering tests, the freeze-thaw behavior of fines in Fines Pond #1 was also studied in the winter of 1991–92. Water was removed from the top of the pond and the fines/sand layer was exposed to winter weather. Following the thaw in the spring of 1992, it was observed that the solid contents of the settled fines had increased to greater than 48 weight percent (Figure 2-9). It was surmised that freeze and thawing contributed significantly to the increase of solids content as compared to the effect of sand loading alone.

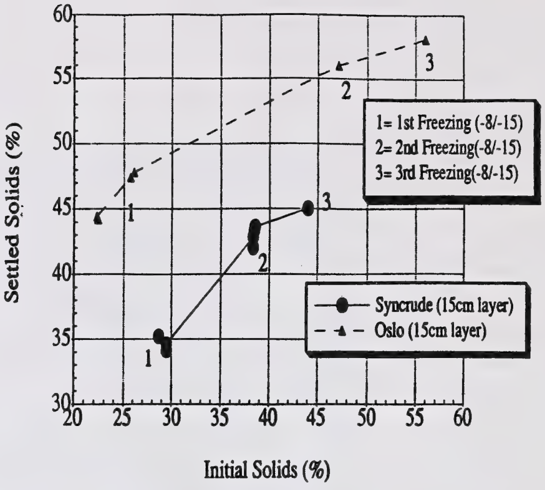

FIGURE 2-9. Effect of freeze thaw, multi-cycle on solids content, OCWE and CHWE processes (initial solids *versus* settled solids).

In parallel with the above test program on Lease 41, layering of various thicknesses of fines was also studied in large sized (3 m diameter × 3 m high) tanks. The greatest thickness of fines that was layered with sand was 2 metres. In this particular test, the fines had an initial solids content of

TABLE 2-2. Consolidation of fine tails at Lease 41 — Pond 1.

Sampling period	Elevation	Sample number (r)	Solids %	% passing 22 μ	CMW-R	FW-R	Comments
July 24/91	98.620	87	25.09	63.81	0.30	0.47	Closest sample pre-surface collapse.
Nov. 11/91	98.625	311	45.02	56.59	0.41	0.52	
May 92	98.494	16–3	72.52	63.48	1.29	0.71	
June 17/92	98.550	210	66.1	61.76	1.01	0.66	SAME RELATIVE POSITION
July 15/92	98.400	233	66.52	63.13	1.29	0.67	
July 24/91	98.470	88	28.92	62.5	0.22	0.48	Closest sample pre-surface collapse.
Nov 11/91	98.455	312	40.28	68.06	0.37	0.54	
May 92	98.375	16–5	62.26	95.28	1.24	0.73	SAME RELATIVE POSITION
July 24/91	98.330	89	30.79	65.03	0.16	0.49	No sample taken in May.
Nov 11/91	98.360	313	46.64	62.01	0.40	0.55	
June 17/92	98.250	211	46.04	97.43	0.61	0.65	SAME RELATIVE POSITION
July 15/92	98.201	334	52.47	84.75	1.04	0.65	

CMW-R: Clay mineral to water ratio, from Methylene Blue Index and solid content analysis (larger number indicate increased consolidation)

FW-R: Fines < 22 micron over fines plus water ratio (similar number indicate no infiltration of coarse sand) only 25 weight percent and sand layering was only partially successful as some toppling of sand was observed. In other tanks, though, where fines were a meter thick or less and the solids content was 28 to 30 percent the sand layered over the fines and no toppling was observed.

In summary, tests on various scales (tank tests, pond tests, *etc.*) indicated that sand layering can work effectively but the sand must be layered carefully and the solids contents of fines must be about 30 percent by weight to begin with or sand toppling may occur. Using the sand buggy to discharge slurried sand by impinging on a spoon provided enough thrust that the buggy was able to move and traverse the length of pond and spray sand with ease and without incurring additional cost. The buggy design should be modified to incorporate two spoons, one on each end of the buggy, so that the buggy can traverse in both directions using back pressure thrust.

OSLO studied the feasibility of a commercial layering system to compare its costs with a wet landscape tailings management system. The results indicated that a solid tailings disposal scheme could be developed and that the costs of tailings management could be substantially reduced. The solid tailings management system may have greater appeal to stakeholders concerned with tailings management in oil sands projects.

2.7 Solids Tailings Management Alternatives

In a commercial operation tailings from the OSLO processes can be managed by using both the freeze-thaw technique and the technique of layering fines (containing 30 weight percent solids) with sand, as devised at Lease 41. The freeze-thaw technique enhances fines densification from 30 percent solids to about 45 to 50 weight percent at which point successful sand layering is assured. Some of the ways these techniques can be combined to achieve consolidation of fines for reclamation are discussed below.

2.7.1 Alternative A

Solidify fines yearly by storing them in a tailings pond to mature to about 25 to 30 percent solids then densify them in a separate pond in thin layers (one to two metres thick) using the freeze-thaw technique. Then place a 0.5 m thick layer of sand over them. Repeat this procedure for successive layers.

2.7.2 Alternative B

Use two to three ponds instead of one big pond and place total tailings in each pond so that the fines layer doesn't exceed one metre in thickness. Use the freeze-thaw technique to densify fines in each pond in the winter, then use the sand layering technique in each pond during summer. All the ponds can be solidified every year as fines are being generated.

2.7.3 Alternative C

Accumulate all fines, in a fluid state, in one pond until the mine reaches the end of its life, then use the freeze-thaw densification technique in a separate pond. This approach may take years to reclaim the mine site after the cessation of production.

The fines could also be left in a fluid state and capped with water (as in the present commercial project scenario).

2.7.4 Alternative D

Accumulate all fines in fluid state in one pond but keep the dykes stepping inwards into the pond so that the toe of advancing beaches keeps burying fines that have reached a density of about 30 weight percent solids. Fines with 30 percent solids have enough strength that any sand raining from beaches on to them will form a layer over the fines thus burying them. In this way a pond can be created such that at the end of project a minimal amount of fines are left. These fines could then be solidified using the freeze-thaw and sand layering techniques described earlier.

A cost/benefit study should be carried out for all the above alternatives to determine the best method for an oil sands mine.

2.8 Discussion of Results and Conclusions

The following conclusions are drawn from the OCWE/OHWE process development programs:

- Both the OCWE and OHWE processes generate fine tails that have superior settling, consolidation, and strength characteristics when compared with tailings from the CHWE process. These properties enable the application of solid tailings management techniques to substantially reduce the volume of fine tails. OSLO studies also indicate a substantial reduction in tailings management costs for solid tailings management systems as compared to the present fluid tailings management system.
- Layering sand over settled OCWE fines to significantly enhance consolidation was demonstrated both in laboratory and field tests on Lease 41. A unique system of inter-layering fines and sand was demonstrated on oil sands for the first time on Lease 41. This has the potential to be a viable, low cost tailing management system.

- The 1994 comparison of the OHWE and CHWE processes sponsored by the Fine Tails Consortium indicated that OHWE fine tailings contain a lesser amount of <2 micron size clay particles than CHWE fine tailings. These are the particles that are considered to be the mainly responsible for the formation of fluid fine tails that require containment.
- The determination of PSD for fines was performed with the standard ASTM procedure in which fines are dispersed with Calgonite. OSLO tests used distilled water instead of Calgonite without knowing that it too disperses fines. In 1994 Dr. A. Sethi of MRRT pointed out that the dispersing effect of distilled water was the reason for the similarity of PSDs of both OHWE and CHWE fines. The PSD of fine tailings from various processes must be carried out in such a way that clay aggregates are not intentionally dispersed. It is recommended that the Methylene Blue Index and double hydrometer techniques developed by Suncor/MRRT be used rather than the standard ASTM procedure.
- A predictive consolidation model for OCWE fines has been developed by Golder Associates Ltd. of Calgary and has proven to be a good tool for the Lease 41 layering test. This model requires further validation as the OCWE process proceeds to commercialization.
- Both the processes are considered ready for testing at oil sand processing rates of 1000 to 4000 tph. Test work at this scale is thought to be necessary before commercial application.

2.9 Acknowledgments

The author would like to thank OSLO Joint Venture Owners and AOSTRA for allowing this information to be included in the Sludge Consortium's Information Volumes. It is recognized that both OCWE and OHWE are patented processes and that the OHWE process technology was offered for comparison test work only. It is not a part of the Fine Tails Fundamentals Consortium's technologies.

2.10 References

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3.0 A COMPARISON OF TAILINGS FROM OSLO AND CLARK HOT WATER EXTRACTION PROCESSES

3.1 Laboratory-Scale Experiments

3.1.1 Introduction

The Clark Hot Water Extraction process for the recovery of bitumen from oil sands produces fine tailings that are slow settling and somewhat toxic. Over time, the fine tailings settle to give a clear water layer and a mature fine tails phase with about 30% solids by weight which is difficult to compact further. An alternative process developed by the OSLO group [1–3] and known as the OSLO Hot Water Extraction (OHWE) process is reported to produce tailings that settle quickly, are non-toxic, and give a mature fine tails phase which is easy to compact further.

The OHWE process uses kerosene and methyl isobutyl carbinol (MIBC) as process aids instead of the sodium hydroxide that the Clark process uses. It can be operated in the temperature range of 30°C to 80°C but requires the addition of air to the slurry. Air is injected directly into the pipeline that carries slurried oil sand to the separation vessels. This differs from the Clark process in which aeration occurs naturally as oil sand is being conditioned in a tumbler with hot water. With minor modifications, the OHWE process can be operated in equipment designed for the Clark process.

The laboratory work reported here was undertaken to compare the two processes on Suncor and Syncrude ores and to attempt to explain the differences in the behavior of tailings from the two processes.

3.1.2 Laboratory Experiments Using the OHWE Process

Eighteen oil sands were processed using a laboratory-scale version of the OHWE process supplied by the OSLO Consortium. The experiments were carried out in Syncrude's standard Batch Extraction Unit [4] using water from the Athabasca River.

Samples of all of the oil sands used in this study were taken shortly after they were mined. Some samples were processed almost immediately while the rest were stored at –30°C until needed. Storage at –30°C helps to eliminate aging effects [5,6]. Some of the oil sands were obtained from the Syncrude lease and some from the Suncor lease.

Tailings samples from the experiments were allowed to cool to room temperature then subsampled. Each subsample was centrifuged (20 minutes at 20,000 rpm) then the clear supernatant was decanted and a portion reserved for the determination of toxicity, electrophoretic mobility (EM), and anions (chloride, sulfate, and bicarbonate). A second portion of the supernatant was ultrafiltered and the ultrafiltrate analyzed for cations (iron, aluminum, magnesium, sodium, calcium, and potassium). All the tailings remaining after removal of the subsample were transferred to graduated glass cylinders which were sealed with polyfilm. The position of the interface that developed in these cylinders was monitored over the course of the next year.

A Rank Brothers electrophoresis apparatus was used to measure the electrophoretic mobility of fine solids in the aqueous phase of the original tailings. A rectangular cell was used and particle mobilities were measured at 25°C at both of the stationary planes.

Toxicity was measured on centrifuged tailing samples using the method described in Ribo and Kaiser [7]. This method measures the Inhibition Concentration of the test sample which causes a 50% reduction in light emission by the exposed photobacterium phosphoreum over a 15-minute time period (IC_{50}). An IC_{50} of 20 means that a 20% concentration of the test sample resulted in a 50% decrease in the relative light emission. An IC_{50} of 100 means that the neat test sample did not result in a 50% decrease in relative light emissions. A high IC_{50} result means a high concentration of test sample was needed to produce a 50% decrease in light; thus, the higher the IC_{50} the lower the toxicity.

Settling curves (sediment volume *versus* elapsed time) were plotted for all experiments, and the curves were used to determine the time needed to reach 50% settled as well as the sediment volume after one year. Throughout this paper, sediment volume after one year is referred to as final sediment volume because in all cases settling had virtually ceased.

Descriptions of our experimental procedures have already been published [8,9] as have anion and cation concentrations and data related to settling, toxicity, and electrophoretic mobility. The settling rate (days to 50% settled) for OHWE tailings was highly variable. In some cases it was very fast (<1 day) and in other cases very slow (>100 days). This was surprising because the OHWE process was expected to produce tailings which would invariably settle in less than a day. Five of the oil sands did produce tailings which settled in less than a day, but the other 13 produced tailings with settling rates similar to those usually associated with tailings from the Clark process.

Toxicity results were also variable, ranging from almost non-toxic to highly toxic. Again this result was surprising because the OHWE process was expected to produce non-toxic tailings. Tailings from five of the oil sands were relatively non-toxic, but the other 13 produced tailings with toxicities similar to those usually seen with tailings from the Clark process. Electrophoretic mobilities were also highly variable (–2 to –4 μm/s/V/cm), as were final sediment volumes (370 to 750 mL/kg oil sand).

3.1.3 Explanation for Low Settling Rate and High Toxicity

A plot of settling rate *versus* electrophoretic mobility for the OHWE tailings is shown in Figure 3-1. Settling rates were very fast (<1 day) when the electrophoretic mobility was low and slow when the electrophoretic mobility was high. Also, settling rate appeared to be related to the concentration of calcium ions in the aqueous phase of the tailings (Figure 3-2). Tailings with high concentrations (>7 ppm) of calcium settled fast (<1 day) and tailings with low concentrations (<7 ppm) settled slowly.

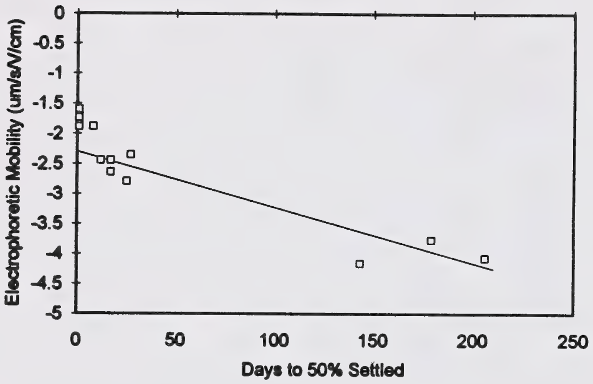

FIGURE 3-1. Effect of EM on settling rate (OHWE tails).

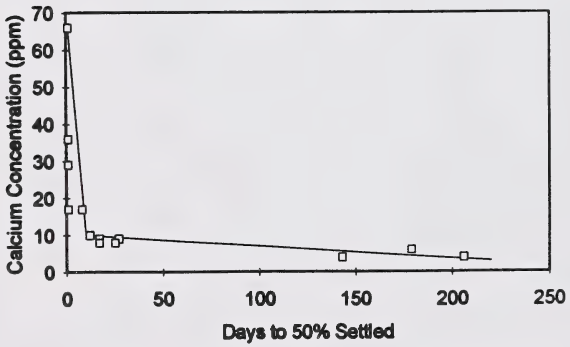

FIGURE 3-2. Effect Ca on settling rate (OHWE tails).

It has been shown [10,11] that the electrophoretic mobility of (therefore the charge on) particles suspended in fine tailings from the Clark process is correlated with the concentration of natural surfactants in those tailings. The higher the concentration of natural surfactants in the tailings, the more negative the charge on the fine particles. The current work has demonstrated (Figure 3-1) that the settling rate of particles suspended in fine tailings from the OHWE process is also related to particle charge, the more negative the charge the slower the settling rate.

The existence of a relationship between settling rate and charge, and between charge and surfactant concentration, suggests that it is the concentration of natural surfactants in tailings that controls the settling rate. It is proposed that if the concentration of natural surfactants in a tailings system is high then the fine particles in that system will be covered with adsorbed surfactant molecules and will, as a result, be both highly charged (negatively) and bulky (a better word might be fuzzy). A combination of electrostatic and steric effects would be expected to result in slow settling.

Because the OHWE process does not use sodium hydroxide, one might expect that natural surfactants would be either absent from OHWE tailings or present in low concentrations. However, available information indicates that this supposition is not correct. Several studies [10] on the effect of sodium hydroxide on surfactant concentrations in tailings have included extractions with de-ionized water and no sodium hydroxide. Many ores gave relatively high surfactant concentrations without sodium hydroxide. However, there were also ores that gave very low natural surfactants concentrations when extracted with water. Therefore, although the OHWE process does not use sodium hydroxide it is expected that many ores processed this way will produce tailings containing natural surfactants. So it is not surprising that the OHWE process can sometimes produce slow settling tailings.

The finding (Figure 3-2) that the settling rate of OHWE fine tailings can be related to the concentration of calcium in the aqueous phase supports the theory that slow settling is due to the presence of natural surfactants. According to the literature [12,13] the natural surfactants are sodium naphthenates and sodium sulfonates. These would be expected to react with calcium compounds to form insoluble (hence inactive) calcium salts. Tailings with high calcium concentrations should therefore have low natural surfactant concentrations and be fast settling. This indeed is what was found. Tailings with calcium concentrations above about 7 ppm settled to the 50% point in less than a day, while tailings with calcium concentrations below 7 ppm settled slowly.

Toxicity of the aqueous phase of tailings was also related to calcium concentration. Figure 3-3 relates toxicity (IC_{50}) to calcium concentration. Tailings were almost always non-toxic below about 7 ppm of calcium and almost always toxic above 7 ppm of calcium.

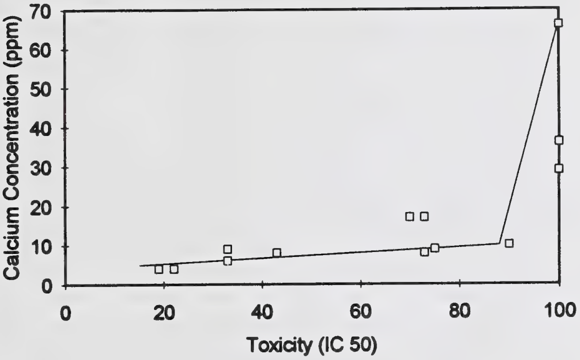

FIGURE 3-3. Effect Ca on IC_{50} (OHWE tails).

The literature [14] attributes tailings toxicity to the presence of naphthenates. Since in theory high concentrations of calcium are consistent with low naphthenate concentrations, it would be expected that tailings with high measured calcium concentrations would have low measured naphthenate concentrations and would therefore be non-toxic. Likewise, tailings with low measured calcium concentrations would be expected to be toxic. This *a priori* argument is in accord with experimental measurements (Figure 3-3). Tailings with high calcium concentrations (>7 ppm) were low in toxicity and tailings with low calcium concentrations (<7 ppm) tended to be high in toxicity.

Experiments we performed with the OHWE process used Athabasca River water containing 60 ppm of calcium. This water is evidently the source of the calcium which finally appears in tailings. Since the tailings, in all cases, had less than 60 ppm of calcium the system evidently consumes calcium. Differences in settling rate do not appear to be related to the concentration of bicarbonate in the aqueous phase because there was almost no difference in the amount of bicarbonate found in the various tailings samples.

In the final analysis, the properties of tailings produced by the OHWE process using river water appear to be dependent on the oil sand being extracted. It could also be said that oil sands which produced tailings with more than about 7 ppm of calcium produced tailings that were fast settling and non-toxic, while oil sands which produced tailings with less that about 7 ppm of calcium produced tailings that were slow settling and toxic. As a general rule Suncor oil sands produced fast settling, non-toxic tailings, while Syncrude oil sands produced slow settling, toxic tailings. One should keep in mind that the number of oil sands in the study was limited, thus making it difficult to generalize the results.

3.1.4 Laboratory Experiments on the Clark Process

Eight oil sands were processed using the standard laboratory version of the Clark process [4]. These oil sands were a subset of the oil sands used in the OHWE experiment. This subset consisted exclusively of Syncrude oil sands which (with one exception) produced slow settling, toxic tailings when extracted with the OHWE process. The experiments were carried out to obtain data with which to compare the OHWE process and the Clark process on typical Syncrude ores. The oil sands were processed with water from Syncrude's recycle pond using 0.02 wt% sodium hydroxide. Experience has shown that this dosage is sufficient to produce a primary recovery close to the maximum for oil sands with 8 to 11% bitumen.

Data available [8,9] from these experiments include anion and cation concentrations in the aqueous phase of the tailings as well as settling rates, toxicities, and electrophoretic mobilities.

Tailings from all eight oil sands were slow settling, highly toxic, contained fine particles with high electrophoretic mobilities (more negative than –3 μm/s/V/cm), and had low calcium concentrations (<5 ppm). Because the role of sodium hydroxide in the Clark process is to generate natural surfactants [10], all these tailings would be expected to have high concentrations of natural surfactants. Furthermore, calcium ions were not added during processing because Syncrude's recycle pond water contains little calcium in comparison to river water. According to the theory outlined previously, the high concentrations of natural surfactants in these tailings would be expected to lead to high negative charges on the fine particles (making them slow settling) and high toxicities.

Settling times were compared for the eight Syncrude oil sands extracted with both processes. Keep in mind all of them produced slow settling tailings from OHWE extractions. Settling times averaged 78 days for tailings from OHWE extractions and 81 days for tailings from Clark extractions. For these eight Syncrude oil sands, then, there was no significant difference in fine tailings settling rates for the two processes.

Toxicities of tailings from the same set of oil sands were also compared for the two processes. Toxicities were fairly similar for the two processes, with OHWE tailings being generally a bit less toxic than Clark tailings. However, the advantage of the OHWE process over the Clark process in terms of toxicity was not large.

Final volumes of sediment were also compared, and there was no significant difference between the two processes.

3.1.5 Calcium Sulfate to Improve Tailings Properties

If natural surfactants are responsible for low settling rates and toxicity, it should be possible to convert slow-settling, toxic tails to rapid-settling, non-toxic tails by adding calcium compounds to precipitate the surfactants. Calcium sulfate and calcium hydroxide could be used, but we chose calcium sulfate. Because of the high concentration of bicarbonate, both compounds will convert to calcium carbonate when added to tailings. However, the solubility of calcium carbonate is pH dependent [15] and its solubility decreases with increasing pH. With calcium sulfate we should be able to introduce more calcium into the aqueous phase because calcium sulfate causes only a small change in pH, while calcium hydroxide raises the pH significantly. A source of commercial quantities of surplus calcium sulfate exists at Redwater.

The idea that calcium compounds could act to accelerate settling rates was first tested on OHWE process tailings from oil sand #8801-3 using 130 ppm of calcium sulfate hemihydrate. The time to 50% settled decreased from 35 days to five days. In the end, though, there was no difference in the final volume of sediment.

This success with OHWE process tailings led to an experiment with Clark tailings from the same oil sand (8801-3). The Clark tails were treated with 123 ppm of calcium sulfate hemihydrate. The time to 50% settled decreased from 29 days to nine days and again there was no effect on the final volume of sediment. The bicarbonate concentration in the tailings changed only slightly.

To determine if this result could be duplicated with other oil sands, OHWE tails produced from several more Syncrude oil sands were treated with about 125 ppm of calcium sulfate. Settling rates, final volumes, and water toxicities were determined for the treated and untreated cases and those data are available [16].

The addition of about 125 ppm of calcium sulfate hemihydrate to slow-settling OHWE tailings increased their settling rates. The time to the 50% settled point decreased from an average of 65 days to an average of 11 days. Tailings treated with calcium sulfate also became relatively non-toxic with IC_{50} numbers increasing to 100 in every case. The settling experiments were followed for about 300 days and in most cases settling appeared to have ceased. For each oil sand, final volumes of sediment were the same for the tailings amended with calcium sulfate as they were for untreated tails. However, between oil sands final volumes of sediment were quite different.

Tailings obtained by extracting some Syncrude oil sands with the Clark process were also amended with calcium sulfate hemihydrate and their settling behavior was followed. Settling rates, final sediment volumes, toxicities, and anion and cation concentrations were determined for these tailings [16]. Amendment with 175 ppm of calcium sulfate succeeded in increasing the settling rates from an average of 74 days (to 50% sediment volume) to an average of 18 days. Calcium sulfate had no noticeable effect on toxicity as far as could be determined from the IC_{50} values. Nor was there any difference in final volumes of sediment between the amended and unamended cases. Calcium sulfate did cause a small reduction in the concentration of bicarbonate in the tailings, but there was no measurable increase in calcium concentration.

Failure to reduce toxicity by adding calcium sulfate to process tails was disappointing, and another experiment was conducted to test the effect of higher concentrations. Clark process tailings from oil sand 8806 were treated with 250 – 1000 ppm of calcium sulfate and the tailings waters from the treatments were clarified by either centrifugation or a combination of centrifugation and ultrafiltration. Clarified tailings were analyzed for anions, cations, and toxicity and the settling behavior was measured. These data are available [16]. Addition of 750 ppm of calcium sulfate hemihydrate was required to reduce the IC_{50} to 100. All dosages higher 250 ppm reduced the time to 50% settled from 100 days to four days. There was little, if any, effect on the final volume of sediment over the untreated case. The bicarbonate concentration was significantly reduced, and calcium concentration increased by only a small amount.

An experiment was carried out to determine if treated tailings might be easier to consolidate by mechanical methods such as centrifugation. Treated tailings and a control quantity of untreated tailings were centrifuged in small tubes at 1000 rpm for 30 minutes after which time light transmittance was measured. The tubes were then centrifuged at 2000, 4000, 6000, 8000, and 10,000 rpm, with light transmittance being measured after each centrifugation. These data have already been published [16]. The treated tailings were very easy to consolidate by centrifuging, with 16.1% of light passing after the 2000 rpm centrifugation. Essentially all the solids were contained in a compact sediment in the bottom of the tube. On the other hand, untreated tailings required centrifuging at 10,000 rpm before light would pass through the supernatant and the solids would form a sediment in the bottom of the tube. Clearly, calcium sulfate treated tailings are much easier to compact by centrifugation than untreated tailings.

3.1.6 Treating Water to Improve Tailings Properties

During the course of this study it was suggested [17] that the production of slow settling, toxic tailings from the OHWE process could be eliminated by reducing the level of bicarbonate in process water. It was proposed that bicarbonate could be removed from either pond or river water by treatment with sulfuric acid followed by lime. Suncor treated a sample of Syncrude pond water, and Syncrude treated a sample of river water following directions provided by Suncor. Analytical data for Syncrude pond water, river water (summer and winter), treated pond water, and treated river water are given in Table 3-1.

Treated pond water contained less bicarbonate and more calcium than untreated pond water, likewise for treated and untreated river water.

Eight oil sands were extracted in the laboratory using the OHWE process and treated pond water. Four were Syncrude oil sands which had been stored in the freezer at –30°C. The other four oil sands were freshly mined and homogenized by Suncor and stored in the frozen state. They were shipped frozen and on arrival in Edmonton were placed in –30°C storage. At the time they were processed these oil sands had been in frozen storage for only three to four weeks compared to three to four years for the Syncrude oil sands.

For each experiment, primary and secondary recoveries were determined and the settling behaviors of the tailings were monitored over time. In addition, the aqueous phases of the tailings were analyzed for anions and cations and toxicities were determined [18].

The combination of the OHWE process and treated pond water produced tailings which in all cases settled quickly (<1 day) and were less toxic than Clark tailings but more toxic than OHWE tailings from untreated river water. The final volume of sediment was about the same as that obtained from the OHWE process using untreated river water and from the Clark process using untreated pond water.

For the four Syncrude oil sands, the use of treated pond water resulted in tailings that settled faster and were less toxic than tailings from untreated river water. For the four Suncor oil sands treated water made little difference. They all produced fast settling, non-toxic tailings even with untreated river water.

The theory already presented suggests that it is the concentration of natural surfactants in tailings that controls settling rate and toxicity. If the natural surfactant content of a tailings sample is high, then the fine particles will be highly charged (negatively) and electrostatic repulsion between the particles will cause slow settling. The tailings will also be toxic because natural surfactants are in themselves toxic. One way to reduce the content of natural surfactants is to add calcium ions which we believe will react with naphthenates to form insoluble calcium derivatives. Accordingly it may be the high calcium content of treated pond water that is responsible for the faster settling rate, less toxic tailings.

TABLE 3-1. Water analysis.

Species	River water (summer)	River water (winter)	Syncrude pond water	Treated pond water	Treated river water
Cl^- (ppm)	6	14	136	50	16
SO_4^{--} (ppm)	26	37	215	1227	153
HCO_3^- (meq/L)	2.2	3.3	14.2	4.3	0.4
Mg^{++} (ppm)	10	17	4	6	9
Ca^{++} (ppm)	35	60	5	160	44
Na^+ (ppm)	18	–	538	421	18
pH	7.7	7.7	7.7	7.9	–

3.1.7 Conclusions

- The OHWE process, for selected oil sands, produced fine tailings which were fast settling and non-toxic. Other feeds produced slow settling, toxic tailings.
- Ores which produced tailings with high concentrations of natural surfactants were the ones that produced slow settling, toxic tailings.
- Slow settling, toxic tailings from the Clark process can be converted to fast settling, non-toxic, easily compacted tailings by adding calcium sulfate to them.
- Ores which, when extracted with the OHWE process and untreated river water, give slow settling, toxic tailings will give fast settling, non-toxic tailings by extracting them, instead, with water that has been treated with sulfuric acid followed by lime.

3.1.8 Recommendations

The laboratory procedures used in this study required considerably more water, for both processes, than would be used commercially. Consequently, the findings need to be confirmed by continuous pilot-scale runs using ores that have been selected to produce both slow and fast settling tailings.

3.1.9 Acknowledgment

The author would like to thank OSLO Joint Venture owners and AOSTRA for allowing this information to be included in the Fine Tails Consortium's Information Volumes. It is recognized that OCWE and OHWE are patented processes and that the OHWE process technology was offered for comparison test work only. It is not a part of the Fine Tails Fundamentals Consortium's technologies.

3.1.10 References

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3.2 Pilot Tests

3.2.1 Abstract

The OSLO and Clark Hot Water Extraction processes were compared with respect to bitumen extraction performance and tailings characteristics using Syncrude Research's Experimental Extraction Circuit pilot plant. Each process was run under optimum conditions on two different oil sands, one assaying 12.4% bitumen from the Syncrude mine and another assaying 12.3% bitumen from the Suncor mine.

The Clark process used recycle water from Syncrude's Mildred Lake Settling Basin (MLSB), while treated Athabasca River water was used for the OSLO process. For the OSLO process only, one additional run was made with untreated Athabasca River water on Syncrude oil sand to determine the effect of water treatment.

Both processes exhibited similar bitumen recoveries and froth qualities with each oil sand, and water treatment had no effect on the extraction performance of the OSLO process.

Initially, OSLO fine tails settled faster than Clark fine tails. However, the settling curves converge with time so that after 500 days the settled volume of OSLO fine tails is only 10% less than that of Clark fine tails. Given sufficient time, final volumes are expected to be about the same. Addition of calcium sulfate to fresh Clark tails resulted in settling behavior very similar to that of OSLO tails generated from treated river water.

3.2.2 Introduction

The Clark Hot Water Extraction (CHWE) process used at the Suncor and Syncrude plants in northern Alberta mixes oil sand with hot water and steam to create an 80°C slurry from which bitumen is readily separated in gravity settling and induced air flotation equipment. A small amount of sodium hydroxide is used during tumbling to aid in bitumen flotation.

The OSLO Hot Water Extraction (OHWE) process [1,2] is similar except that it uses kerosene and methyl isobutyl carbinol (MIBC) rather than sodium hydroxide. In addition it requires the introduction of air into the oil sand slurry and is reported to be operable down to 30°C whereas the CHWE process is not operable below about 45°C.

Fine tails produced by the OHWE process are reported [2,3] to have superior properties such as reduced volume, faster dewatering, and higher strength. Higher strength would permit them to be surcharged with sand, which would further accelerate dewatering. These differences, if demonstrated, would suggest that the OHWE process would result in a smaller accumulated volume of mature fine tails (MFT) over the life of an oil sands project. Barring any process disadvantages this could create the economic incentive needed to consider retrofitting the OHWE process into existing operations.

The principal objective of this program was to compare the properties of tailings from the OHWE and CHWE processes. To do this, large quantities of oil sand needed to be processed in a pilot plant under conditions that were optimal for each process. This provided an opportunity to compare each process with respect to bitumen recovery, froth quality, and the distribution of fine solids in process streams, as well as to determine the effect of water treatment on the OHWE process.

These two processes were compared on the Experimental Extraction Circuit (EEC) pilot plant at the Syncrude Research Center using two different oil sands — one from the Suncor mine and one from Syncrude's mine. Recycle water from Syncrude's Mildred Lake settling basin (tailings pond) was used for the CHWE runs, while treated Athabasca River water was used for the OHWE runs. Suncor's research [3,4] had indicated that properties of tails from the OHWE process were insensitive to oil sand quality if the process water was first treated with sulfuric acid and lime. To determine the effect of water treatment on the OHWE process, an additional run was made using Syncrude oil sand and untreated Athabasca River water.

Settling tests were conducted on the fine-grained fraction of the tailings. As well, both coarse-grained and fine-grained tailings fractions were characterized chemically.

This pilot test program was a joint effort between Syncrude, Suncor, Imperial Oil Resources Ltd., the OSLO Consortium, and the Fine Tails Fundamentals Consortium, including CANMET, the Alberta Research Council (ARC), the National Research Council (NRC), and the University of Alberta.

3.2.3 Summary of Extraction Performance — Experimental Program

3.2.3.1 *Pilot Plant Description*

The flow sheet for Syncrude's EEC pilot plant is shown in Figure 3-4. The main process vessels used were the Tumbler, Primary Separation Vessel, and Secondary Flotation unit.

Oil sand to the tumbler was screened through a grizzly with 10.2 cm × 10.2 cm openings. The tumbler itself was 152 cm long with an inside diameter of 105.7 cm and an exit weir height of 15.2 cm. These dimensions provided the slurry with a residence time in the tumbler of about 4.4 minutes. Internals included two rock ejectors and two paddles that were 27.9 cm high with seven fingers on each paddle for agitating the slurry. Process aids were added with the tumbler water, except for some of the OHWE runs where kerosene and MIBC were also added to the flooded slurry pump box. For all OHWE runs, MIBC and kerosene were used in a volumetric ratio of one part MIBC to two parts kerosene.

Slurry leaving the tumbler passed through a double deck screen to remove oversize material. Screen openings were 2.54 cm × 2.54 cm for the upper deck and 0.64 cm × 0.64 cm for the lower deck. Slurry passing through the screen entered a pump box into which flood water was added to facilitate the subsequent separation of bitumen from solids.

Flooded slurry from the pump box passed through a section of pipe 18.3 m long × 2.54 cm in diameter before entering the Primary Separation Vessel (PSV). For OHWE process runs only, flooded slurry was aerated [5] as it entered this pipeline. Slurry residence time in the line was approximately 10 seconds.

The PSV has a transparent glass wall 0.61 m in diameter × 2.26 m high with a rake in the bottom cone for desanding. Froth overflowed into a launder and then into weigh tanks mounted on load cells for accurate rate measurement. As shown in Figure 3-4, middlings was pumped to a secondary flotation unit which was a two-cell Outokumpu induced air flotation machine equipped with mechanical agitators. Each of its two interconnected cells is 0.4 m long × 0.4 m wide × 0.5 m deep. Lean froth flowed by gravity from the flotation machine to a 0.4 m diameter froth cleaner from which the final secondary froth product flowed into a pair of weigh tanks. Tailings from the PSV and flotation machine were combined and pumped to an experimental flume described in Miller *et al.* [6].

Recycle water from Syncrude's tailings settling basin was stored in a 30.3 cubic meter indoor tank adjacent to the EEC pilot plant. Athabasca River water was stored in a 113.6 cubic meter outdoor tank equipped with a recirculation loop and pump. A metering pump was used to add acid or lime to the recirculation line.

The pilot plant was equipped with instruments that measured flow rate, density and temperature of most process streams. Complete operating data and performance measurements are given in the report by Lowe, Spence, and Sury [5].

3.2.3.2 *Oil Sands Description*

Table 3-2 provides a brief physical description of the two oil sands used in this program. However, a more complete description is provided by Cuddy [7].

3.2.3.3 *Program Description*

Table 3-3 shows the various combinations of oil sand, water, and extraction process that were chosen for each of the five two-day runs we conducted. For each process, the first day of running was used to determine operating conditions that would maximize bitumen recovery consistent with good froth quality. The parameters that were manipulated were chemical addition rate, water-to-ore ratio, and (for the OHWE process) air addition rate.

TABLE 3-3. Experimental conditions.

Oil sand	Process	Water
Syncrude	OHWE	Untreated Athabasca River
Syncrude	OHWE	Treated Athabasca River
Syncrude	CHWE	Pond recycle
Suncor	CHWE	Pond recycle
Suncor	OHWE	Treated Athabasca River

TABLE 3-2. Description of oil sands.

Oil sand	Suncor 9311B	Syncrude 9310
Bitumen (wt%)	12.3	12.4
Water (wt%)	4.7	4.3
Solids (wt%)	83.0	83.3
Fines content (wt% <44 m) (Syncrude XDC method)	13.2	18.5
Geology	Lower estuarine tidal channel-tidal flat	Subtidal (channel-fill) and inter-tidal estuarine

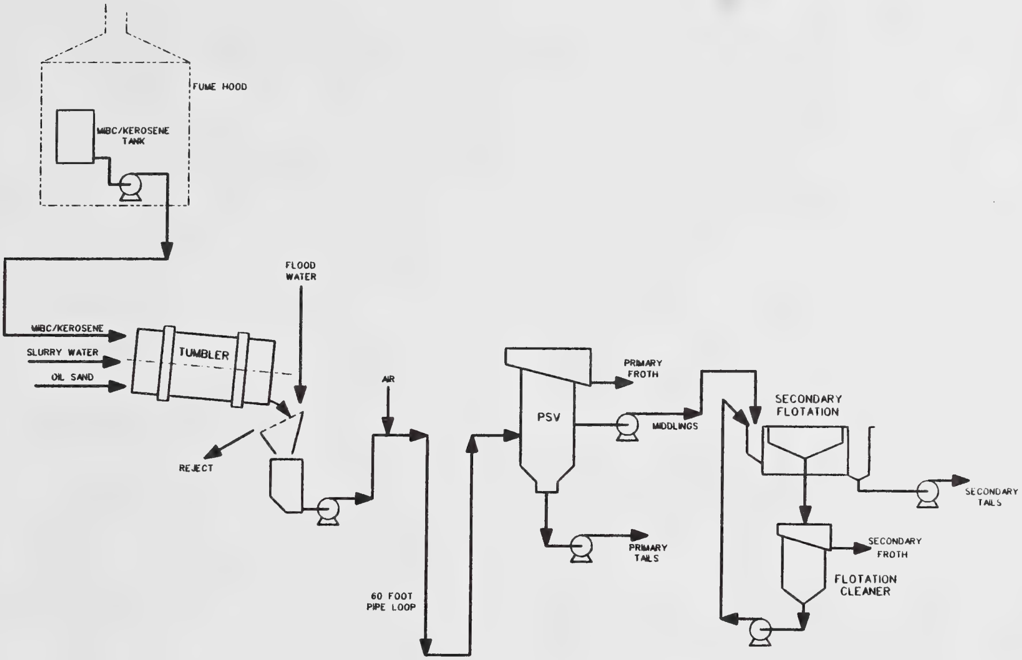

FIGURE 3-4. EEC flow diagram for OHWE runs.

TABLE 3-4. Optimized operating conditions.

Process	OHWE	OHWE	CHWE	CHWE	OHWE
Oil sand source	Syncrude	Syncrude	Syncrude	Suncor	Suncor
Oil sand ID	9310	9310	9310	9311B	9311B
Oil sand rate (g/s)	694	694	694	694	694
Water type	Athabasca River untreated	Athabasca River treated	Recycle	Recycle	Athabasca River treated
Target tumbler water (wt%)	25	25	25	30	30
Target tumbler slurry temp (°C)	80	80	80	80	80
Target total water (wt%)	65	65	65	55	70
Target flooded slurry temp (°C)	80	80	80	80	80
Secondary flotation (rpm)	800	800	700	700	800
Secondary flotation air (SCFM)	3.75	3.75	2	2	2
NaOH (wt% on oil sand)	0	0	0.05	0.005	0
Tumbler: kerosene + MIBC (ppm on oil sand)	375	375	0	0	260
Pump box: kerosene + MIBC (ppm on oil sand)	0	0	0	0	100
Flooded slurry air addition rate (SCFM)	0.5	0.5	0	0	0.55
Flooded slurry air addition pressure (psi)	35	35	N/A	N/A	65

The optimal conditions are listed in Table 3-4. Three mass balances were performed for each of these sets of conditions.

3.2.3.4 *Sampling, Data Collection, and Analysis*

Streams sampled for mass balance purposes included oil sand, oversize reject, flooded slurry, PSV froth, PSV middlings, PSV tailings, secondary froth, and secondary tailings. Flow rates were also measured for these streams. Using stream assays and flow rates we were able to conduct three mass balances on each process run day under essentially the same operating conditions. Mass balance calculations were done with the MATBAL program [8] which uses an error model described elsewhere [5].

Samples were analyzed for oil/water/solids content by the Modified Dean Stark technique. Solids remaining after soxhlet extraction were sent to McMurray Resources Research and Testing (MRRT) for determination of fines less than 44 microns by the wet sieve method.

Chemical characteristics of the process waters and the oil sand connate water are reported elsewhere [9]. PSV and secondary froths were sampled and characterized physically and chemically by a variety of methods. ARC, NRC, and CANMET performed this work and the results are presented in separate reports [10–12].

3.2.3.5 *Water Description and Treatment*

Untreated Athabasca River water, treated Athabasca River water, and recycle water from Syncrude's tailings settling basin were used during the program. Untreated Athabasca River water was collected from Mildred Lake and trucked to the Syncrude Research Center. Syncrude recycle water was trucked to Edmonton from the Mildred Lake plant site and stored in a separate tank.

When treated water was needed, 10% sulfuric acid was added to untreated water over a period of a day to lower its pH from 7.4 to 5.0. The following day a slurry of 4 wt% lime in water was added to the water in the storage tank until its pH once again reached 7.4. The treated water was then ready for use. However, its pH was monitored throughout the program to ensure that it remained relatively constant. Addition of both acid and lime was carried out while recirculating water in the storage tank.

TABLE 3-5. Extraction performance for Syncrude oil sand (average of three mass balances).

Process	OHWE	OHWE	CHWE
Water source	Untreated Athabasca River	Treated Athabasca River	Recycle
Primary bitumen recovery (%)	79.8	78.6	91.2
Secondary bitumen recovery (%)	12.5	13.0	2.0
Combined bitumen recovery (%)	92.3	91.6	93.2
Primary froth			
bitumen (wt%)	71.1	69.6	65.6
water (wt%)	22.3	23.4	24.5
solids (wt%)	6.6	7.0	9.9
density (g/cc	0.62	0.63	0.92
Combined froth			
bitumen (wt%)	63.2	62.3	65.0
water (wt%)	28.6	29.3	24.9
solids (wt%)	8.2	8.4	10.1
Combined tailings			
bitumen (wt%)	0.6	0.6	0.5
water (wt%)	44.8	45.7	46.6
solids (wt%)	54.6	53.7	52.9

TABLE 3-6. Extraction performance for Suncor oil sand (average of three mass balances).

Process	OHWE	CHWE
Water	Treated Athabasca River	Recycle
Primary bitumen recovery (%)	82.0	92.5
Secondary bitumen recovery (%)	11.4	1.9
Combined bitumen recovery (%)	93.4	94.4
Primary froth		
bitumen (wt%)	67.4	64.7
water (wt%)	26.8	28.8
solids (wt%)	5.8	6.5
density (g/cc)	0.66	0.82
Combined froth		
bitumen (wt%)	61.8	63.8
water (wt%)	31.5	29.3
solids (wt%)	6.7	6.9
Combined tailings		
bitumen (wt%)	0.5	0.5
water (wt%)	46.7	42.4
solids (wt%)	52.8	57.1

3.2.4 Results and Discussion

Complete experimental data and mass balances are reported elsewhere [5]. Key results are summarized in Tables 3-5 and 3-6.

3.2.4.1 *Overall Extraction Performance*

Process performance results for the runs on Syncrude oil sand are summarized in Table 3-5. The numbers given are averages for three mass balances performed under the same conditions. Total bitumen recovery was similar for each process, but the CHWE process recovered more of its bitumen in primary froth and less in secondary froth. Also, because the OHWE process introduces air into flooded slurry, the density of primary froth is much lower for that process –0.62 g/cc *versus* 0.92 g/cc for the CHWE process. Compositions of combined froth and combined tailings were about the same for each process.

Results for the runs on Suncor oil sand are summarized in Table 3-6. Combined bitumen recoveries and froth qualities were the same for both processes. Again the CHWE process recovered more bitumen in primary froth and less in secondary froth, and again the density of OHWE primary froth was much lower than CHWE primary froth –0.66 g/cc *versus* 0.82 g/cc. The slightly higher water content of OHWE combined tailings is due to the fact that a slightly higher water-to-oil sand ratio (0.70:1) was needed to achieve optimal results with the Suncor oil sand than was the case for the CHWE process (0.55:1).

3.2.4.2 *Effect Of Water Chemistry*

For the OHWE process treating Athabasca River water had no noticeable effect on either bitumen recovery or froth quality with the Syncrude oil sand. Differences in the numbers in Table 3-5 are within the experimental error of the Syncrude pilot plant.

3.2.4.3 *Bitumen Distribution in Primary and Secondary Separators*

A significant difference has been noted between the OHWE and CHWE processes with respect to bitumen recovery from the PSV. PSV recovery was about 92% for the CHWE process *versus* 80% for the OHWE process. However, the OHWE process (being flotation based) compensated with a higher second stage recovery (12%) than the CHWE process (2%). Total bitumen recovery was similar for the two processes. This suggests that for the OHWE process PSV recovery efficiency might be increased through better aeration of the flooded slurry.

TABLE 3-7. Fines distribution (average of three mass balances).

Process	OHWE	OHWE	CHWE	CHWE	OHWE
Oil sand	Syncrude	Syncrude	Syncrude	Suncor	Suncor
Water source	Untreated Athabasca River	Treated Athabasca River	Recycle	Recycle	Treated Athabasca River
Flooded slurry fines to combined froth	11%	11%	8%	9%	11%
Flooded slurry fines to combined tailings	89%	89%	92%	91%	89%

3.2.4.4 *Fines Balance*

Fine solids less than 44 μm (*i.e.*, "fines") were measured using the wet sieve technique and mass balanced with the MATBAL program. Table 3-7 shows how fines in flooded slurry distributed themselves between froth and tailings. Within experimental error fines reporting to combined froth are similar for the OHWE and CHWE processes. Treating the process water had no effect on fines distribution for the OHWE process with the Syncrude oil sand.

3.2.5 Part II: Summary of Tailings Settling Properties — Experimental Program

3.2.5.1 *Sampling of Fine Tailings*

Fine-grained slurry samples were collected in two different ways:

- Combined tailings (*i.e.*, primary and secondary tails) were collected in 20-L plastic pails three times during each run. The samples were allowed to settle under static conditions for 30 minutes during which time bitumen was repeatedly skimmed off the tops of the pails. After 30 minutes, the fine-grained slurry was siphoned from each pail and these slurries blended together for subsequent experimental work.
- Two flume runs were conducted for each extraction pilot run and runoff was collected in a holding tank at the end of the flume. After each flume run, runoff in the tank was poured into 20-L pails the contents of which were blended together.

The settling behavior of fine tails samples collected by each method was very similar. Thus, while all tests were performed on both sets of fine tails samples, only those results pertaining to the runoff samples will be summarized here. As well, characteristics of coarse tailings are not described here. A more detailed description of both fine and coarse tails is available elsewhere [13–16].

3.2.5.2 *Physical-Chemical Characteristics of Fine Tailings*

Samples of fine tails were collected as the settling columns were being filled. Various analyses and tests were performed to determine their physical-chemical characteristics.

Oil, water and solids — The oil/water/solids assays were determined in the Syncrude Research Laboratory by the Modified Dean Stark method using toluene as a solvent. Results are given in Table 3-8. Both the CHWE and OHWE processes generated fine tails (runoff) with similar solids contents (7 – 8 wt%). This permitted comparison of unhindered settling behavior in the columns without having to adjust the fine tails density.

TABLE 3-8. Oil, water, and solids in fine tails.

Test #	Description	Sample #	Oil	Water	Solids
1	OHWE, Syn ore, Unt. Ath. R. water	T1R	0.52	91.63	8.11
2	OHWE, Syn ore, Tr. Ath. R. water	T2R	0.58	91.15	7.72
3	CHWE, Syn ore, MLSB recycle water	T3R	0.46	89.02	8.48
4	CHWE, Sun ore, MLSB recycle water	T4R	0.62	90.15	7.97
5	OHWE, Sun ore, Tr. Ath. R. water	T5R	0.55	92.96	7.02

Particle size distribution — Particle size distributions (PSDs) were determined with different methods in different laboratories. As expected, the different procedures gave somewhat different results. PSDs of the fine tails slurries were initially determined in the Syncrude Research Laboratory using a Microtrac I (Model 7995). PSDs on these same samples were also determined by sieve and hydrometer by McMurray Resources Research Technology (MRRT) Ltd. Laboratory. The MRRT determinations were done on both chemically "dispersed" and "non-dispersed" samples using previously described [14] analytical procedures.

The Microtrac PSD data are shown graphically in Figures 3-5 and 3-6.

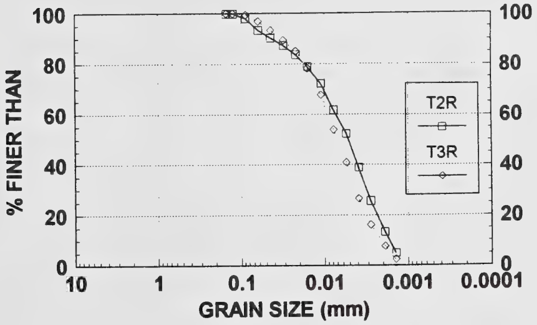

FIGURE 3-5. Particle size distribution by Microtrac — Syncrude fine tails.

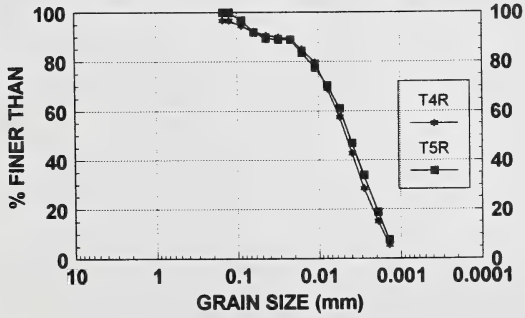

FIGURE 3-6. Particle size distribution by Microtrac — Suncor fine tails.

PSDs by hydrometer are shown in Figures 3-7 and 3-8 for dispersed and non-dispersed samples. Over most of the size range (30 μm – 2 μm) CHWE fine tails are distinguishable from OHWE fine tails by their higher fines content. It is also evident that CHWE tails have reached their limit of dispersion, while OHWE tails can be further dispersed.

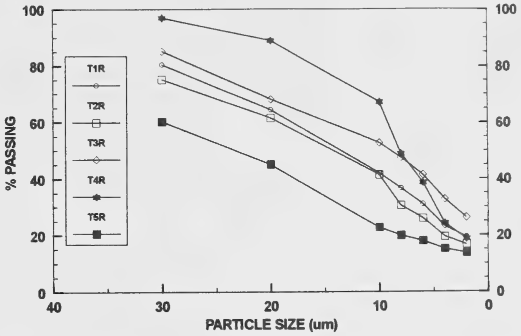

FIGURE 3-7. Particle size distribution by hydrometer — dispersed fine tails.

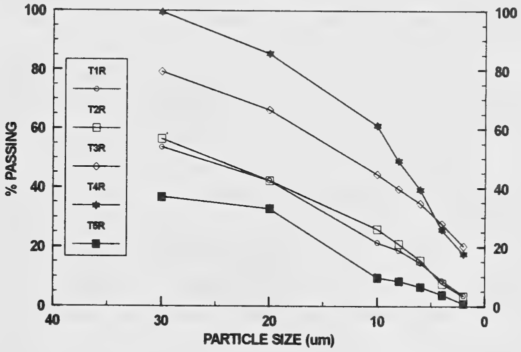

FIGURE 3-8. Particle size distribution by hydrometer — non-dispersed fine tails.

Surface area — After removing bitumen from fine tails, MRRT Ltd. determined their surface area using the methylene blue (MB) adsorption method (Table 3-9). MB adsorption values were determined for both chemically dispersed and non-dispersed solids to assess the degree of dispersion of active clays. OHWE runoff in a non-dispersed state had MB adsorption values of 675 and 692 mL/100 g solids, whereas non-dispersed CHWE runoff had values of 1004 and 940 for Syncrude and Suncor oil sands, respectively. All available data suggest that clays in the CHWE process tails are already quite well dispersed compared to those in OHWE tails — for both oil sands. Consequently, there is a far greater difference in MB adsorption between dispersed and non-dispersed OHWE fine tails than there is for CHWE fine tails.

MRRT Ltd. also determined total surface area (Table 3-9) on bitumen-free samples with a method that uses ethylene glycol monoethyl ether (EGME) as the adsorbed phase [14]. The total surface area ranged between 108 and 140 m^2/g. Suncor fine tails (#4, #5) seem to have a slightly higher total surface area irrespective of the extraction process used.

Mineralogy — As the X-ray diffraction (XRD) data in Tables 3-10 and 3-11 show, the mineralogical characteristics [16] of all of the fine tails are quite similar. Kaolinite ($Al_4Si_4O_{10}[OH]_8$) is the most abundant clay mineral with lesser amounts of illite ($K_xAl_4[Si_{8-x}Al_x]O_{20}[OH]_4$). Some of the illite detected in the total sample (Table 3-10) could be mica as it has a similar diffractogram peak pattern. Minor to trace amounts of mixed-layer clay (illite-smectite) were found in all samples. A description of the meaning of the sample numbers was given in Table 3-7.

The clay fraction (<2 μm) in fine tails as determined by XRD analysis ranges from 29 to 40 percent by weight. The Syncrude runoff fine tails appear to contain a slightly higher amount of clay mineral (81%) than the Suncor runoff fine tails (76%). The composition of the clay fraction in the flume runoff samples suggests that the Syncrude fine tails contains slightly more kaolinite and less illite than the Suncor fine tails in this size fraction.

Initial water chemistry — The water chemistry of fresh OHWE and CHWE fine tails was determined and that of the supernatant waters was monitored over time. Major anions, cations, and pH were determined at the Syncrude Research Laboratory using ICP and IC techniques.

As expected the CHWE fine tailings water had higher sodium and chloride levels than OHWE fine tails [13]. For the OHWE tests, sodium levels in fine tails from Suncor ore were about half those in fine tails from Syncrude ore. Since caustic soda is not used in the OHWE process, this difference likely reflects different levels of sodium salts in the two oil sands. Polyvalent cations were present at relatively low levels in the tailings waters from both ores. Calcium concentrations were quite low (<15 ppm) with the highest levels noted in OHWE fine tails from Suncor ore.

For fresh fine tails, pH values ranged from about 7 to about 8.5. The higher values were more representative of CHWE fine tails.

3.2.5.3 *Settling Tests*

Fine tails were allowed to settle in translucent acrylic columns 14.8 cm in diameter × 122 cm high that were fitted with six evenly spaced sample ports situated between 4.6 cm and 54.6 cm from the bottom. The tubes were filled, in duplicate, to a height of 115 cm in order that one tube could be sacrificed for sampling while the other remained intact to allow the interface between water and sediment to be monitored over time. All tests were conducted at room temperature in the pilot area at the Syncrude Research facilities.

Settling curves expressed as volume % sediment in the columns *versus* time are shown in Figure 3-9. Fresh fine tails start out with 100% sediment. Induction times (time before a water-sediment interface appears) and "final" settled volumes are tabulated in Table 3-12.

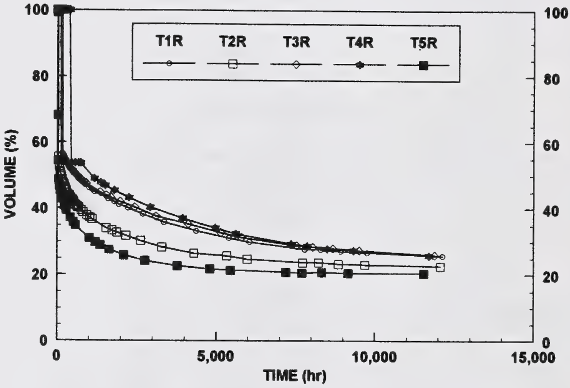

FIGURE 3-9. Settling behaviour of fine tails.

TABLE 3-9. Surface area (MB) and total surface area (EGME) of fine tails.

Test #	Sample #	MB value (non-dispersed)	MB value (dispersed)	% difference in MB values	EGME value (dispersed)
1	T1R	664	1076	62.0	117.6
2	T2R	675	1108	64.1	108.1
3	T3R	1004	1014	1.0	108.1
4	T4R	940	1183	25.8	133.5
5	T5R	692	1122	62.1	139.9

TABLE 3-10. Minerals in fine tails (wt%).

Test #	Sample #	Qtz	Plag	Kfeld	Sid	Kaol	Ill	M-L	Smect
1	T1R	14	0	tr	1	67	17	tr	1
2	T2R	18	1	1	1	63	15	1	0
3	T3R	15	0	1	1	69	13	1	0
4	T4R	19	1	1	3	55	20	1	0
5	T5R	19	1	1	3	55	20	1	0

TABLE 3-11. Minerals in fine tails clay fraction (wt%).

Test #	Sample #	Qtz	Kaol	Ill	M-L	Smect
1	T1R	2	78	15	1	4
2	T2R	2	80	15	3	0
3	T3R	2	81	15	2	0
4	T4R	2	76	18	4	0
5	T5R	2	75	19	4	0

TABLE 3-12. Column settling behaviour of fine tails.

Test #	Sample #	Induction time (hrs)	Settling rate (days)	Volume % (@ 200 days)	Volume % (@ 500 days)
1	T1R	163	22	32	26
2	T2R	16	5	25	23
3	T3R	102	24	32.5	26
4	T4R	448	45	34.5	26
5	T5R	1	2	21.5	20

Induction time — Treating Athabasca River water by acidifying it to pH 5.0 with 10% sulfuric acid and then raising its pH to 7.5 by adding lime greatly reduces induction time for OHWE process tails. The induction time for CHWE fine tails from Suncor ore was much greater than for CHWE fine tails from Syncrude ore. OHWE fine tails from both ores exhibited very short induction times (<1 day).

Settling rate and sediment volume — Settling rate is defined simply as the time it takes fine tails to settle to 50% of their original volume. That is the meaning of the data in Table 3-12. Table 3-12 also reports sediment volumes after 200 and 500 days as a percent of the initial volume of sediment, that is, the volume at time zero. The data show that OHWE fine tails generated with treated water (T2R and T5R) settled faster and to a smaller volume after 500 days than did CHWE fine tails (T3R and T4R). However, as Figure 3-9 shows, the OHWE and CHWE curves are continuing to converge and the CHWE curves have a steeper slope. Thus, in the long term (>two years) there may be little difference between CHWE and OHWE sediment volumes.

If water used for the OHWE process is untreated (T1R), then there is no difference in sediment volume between OHWE and CHWE fine tails. This observation is in agreement with laboratory work [4] conducted by the MRRT Laboratory.

Sediment profiles —Sediment profiles in the columns were monitored and sediment samples were collected from column ports at five different times during the first 36 weeks. These samples were analyzed for oil, water, solids, and PSD as well as for the chemical constituents of the solids and pore water. At each sampling period OHWE sediments had higher concentrations of solids than CHWE sediments. In general, OHWE sediments from the bottom port were coarser than the corresponding CHWE sediments [13].

Water chemistry — Samples of supernatant water were also collected each time sediment samples were taken. They were obtained from either the top port or the port immediately above the mud line. The samples were analyzed for pH, anions, and cations. Generally, pH changed very little with time and pH values for water from CHWE fine tails columns remained higher than those for supernatant water from OHWE fine tails. Anions (chloride, sulfate) and cations also did not change much during the sampling period.

3.2.5.4 *Treatment of CHWE Fine Tails*

Calcium sulfate hemihydrate ($CaSO_4.{}^1/_2H_2O$) was added to fresh CHWE fine tails from both Syncrude and Suncor ores at a dosage rate of 600 mg/L [13]. The addition reduced induction times from 4 days (T3R) and 19 days (T4R) to less than one hour. Very rapid dewatering occurred and dewatering cones (boils) formed on the surface of the sediment as it was contracting. Similar structures were not noticeable in any of the other columns. The time to reach 50% of the original volume decreased from 24 days to 9 days for Syncrude CHWE fine tails and from 45 days to 3 days for Suncor CHWE fine tails.

A comparison of sediment in the columns containing amended CHWE tails and neat CHWE tails indicates that there is a marked increase in the solids content of sediment during the initial settling period. Of note is the fact that calcium sulfate addition acts to increase the suspension of coarser particles [13].

As mentioned earlier, levels of soluble calcium in fine tails were generally low. Amended CHWE fine tails from Suncor ore maintained a level about 55 ppm for at least the first three months [13]. The level for amended CHWE tails from the Syncrude ore, although higher than for CHWE tails, was only about 5 ppm.

3.2.6 Results and Discussion

The solids content, PSD (dispersed), and mineralogy of CHWE and OHWE fine tails were generally similar and this allowed for a good comparison of their settling behaviors. OHWE and CHWE fine tails are distinguishable based on their non-dispersed PSDs and surface areas. CHWE fine tails are more highly dispersed and as a result have higher fines and surface area.

Fine tails from OHWE test runs made with treated Athabasca River water settle faster and to a smaller volume after about 500 days than do fine tails from CHWE test runs. After 500 days the settling curves continue to converge, suggesting that OHWE and CHWE fine tails volumes are likely to be very similar after settling for a few years.

Treating CHWE fine tails with calcium sulfate reduces the amount of clay size fines as well as the surface area. It also reduces induction time and the volume of sediment to almost that of OHWE fine tails.

The difference in settling behavior between amended and neat CHWE fine tails probably relates to the flocculation and agglomeration of clays into domains. The adsorption of calcium ions by the clays reduces the negative charges that are characteristic of clay surfaces, thereby allowing them to flocculate. These structures should have higher permeability than dispersed structures.

3.2.7 Conclusions

- For the two oil sands tested, overall bitumen recovery and froth quality are about the same for the OHWE and CHWE processes.
- The CHWE process has higher first-stage bitumen recovery (92%) than the OHWE process (80%), but the OHWE process compensates with higher second stage recovery (12% *versus* 2%).
- Treating Athabasca River water had no effect on bitumen recovery, froth quality, or fines distribution for the OHWE process.
- CHWE fine tails contain a higher amount of dispersed fines and clay-size particles than OHWE fine tails.
- OHWE fine tails generated with treated river water settled faster than CHWE fine tails generated from recycle water from Syncrude's Mildred Lake Settling Basin.
- OHWE fine tails generated with untreated river water and CHWE fine tails generated recycle water settled at similar rates.
- The CHWE fine tails treated with calcium sulfate hemihydrate settled at rates that were similar to OHWE fine tails generated with treated river water.
- After 500 days settled volumes of fine tails sediment ranged from 20 to 26% and the CHWE fine tails were continuing to settle slowly. All volumes are likely to be similar after a few years of settling.
- Fine tails settling rates are determined by the degree of particle dispersion and by chemistry of the process water.

3.2.8 Acknowledgments

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In addition, the authors thank the OSLO Joint Venture owners for allowing this information to be included in the fine Tails Consortium's Information Volumes. It is recognized that OCWE and OHWE are patented processes and that the OHWE process technology was offered for comparison test work only. It is not a part of the Fine Tails Fundamentals Consortium's technologies.

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4.0 PHYSICO-CHEMICAL AND GEOTECHNICAL ASPECTS OF OHWE FINE TAILING

4.1 Physico-Chemical Aspects of OHWE Fine Tailings

4.1.1 Introduction

Currently, the Clark Hot Water Extraction (CHWE) process is the benchmark for the oil sands industry in terms of bitumen recovery and tailings production. Recently, attention has been focused on perceived problems with tailings accumulation and site reclamation. Consequently, considerable testing has been carried out on alternative bitumen extraction processes to determine whether or not they represent an improvement over the existing technology. The propensity to form fine tailings is a major consideration in the selection of any new process. In particular, the OSLO Hot Water Extraction (OHWE) process has been promoted as being incrementally better than the CHWE process in terms of both lower tailings production and improved physical properties. Preliminary pilot testing indicates that OHWE tailings are non-segregating and have sufficient inherent strength to allow them to be surcharged with coarse sand to increase their solids density.

In order to verify these claims Syncrude and Suncor have collaborated to conduct a series of controlled pilot tests to compare a conventional CHWE operation with two versions of the OHWE process [1]. The testing was carried out at Syncrude's pilot facility near Clover Bar, Edmonton, in the Fall of 1993. The program consisted of a comparison of the following processes: Clark Hot Water Extraction (CHWE), OSLO Hot Water Extraction (OHWE), and a modified OSLO Hot Water Extraction (M-OHWE). Information on the identification and conditions used in the runs are given in Table 4-1. An extensive sampling program was undertaken for each pilot test. Samples of the tailings streams were supplied to both CANMET WRL in Devon, Alberta, and the Institute for Environmental Research and Technology at the National Research Council in Ottawa, Ontario. A list of tailings samples is given in Table 4-2.

CANMET's WRL Laboratory carried out characterization tests on the bulk samples [2,3] while NRC-IERT examined the samples in terms of ultra-fines composition and aggregation [4,5].

4.1.2 CANMET Characterization Tests

4.1.2.1 *Rheology Measurements*

The rheological properties of tailings were measured for suspensions with solids contents ranging to over 50 weight percent. Consolidation of samples was accomplished by pressure filtration. Also, a comparable set of samples prepared from air-dried tailings was tested to determine differences in rheological properties caused by changes in water chemistry.

4.1.2.2 *Index Tests*

Accelerated Settling — Centrifuging at 230 g for only 500 minutes produces MFT sediment volumes that typically are reached after 2 to 500 days of gravity settling.

TABLE 4-1. OHWE-CHWE bitumen extraction runs for Syncrude and Suncor ores.

Abbreviation	Description	Process water
M-OHWE	Modified OSLO hot water extraction	Mildred Lake treated with acid + lime; kerosene + flotation reagent
OHWE	OSLO hot water extraction	Mildred Lake untreated; kerosene + flotation reagent
CHWE	Clark hot water extraction	Pond water + 0.01% NaOH

TABLE 4-2. Tailings stream samples from pilot trials.

Feed ore	Process	PSV middlings	PSV tailings	Secondary tailings	Runoff
Syncrude	M-OHWE	Yes	Yes	Yes	Yes
	OHWE	Yes	Yes	Yes	Yes
	CHWE	Yes	Yes	Yes	Yes
Suncor	CHWE	Yes	Yes	Yes	Yes
	M-OHWE	Yes	Yes	Yes	Yes

Double Hydrometer Tests — These tests measure particle size distribution (PSD) in dispersed and undispersed suspensions. The difference between the percent dispersed and undispersed material for the <2 μm fraction is a quantitative measure of the degree of dispersion. The undispersed PSD correlates with settling behavior and settled volume.

Specific Resistance to Filtration — Water release from a standard suspension volume is measured as a function of time at a specific pressure. Results correlate with settling behavior.

Microscopic Measurement — Aggregate or floc size measurements also correlate with settling behavior.

Electrokinetic Properties — Electrokinetic properties of clay particles correlate with the degree of particle dispersion. The larger the absolute value of the electrophoretic mobility, the greater the degree of dispersion.

4.1.3 Observations

Rheology

- Higher values of elastic modulus (G') and yield point were obtained for fine tailings from the OHWE tests compared to those from the CHWE tests.
- The magnitude of differences between OHWE and CHWE samples were much higher at lower solids contents.
- The absence of measured G' and yield point values for Syncrude and Suncor CHWE fine tailings below 26 and 21 weight percent solids, respectively, indicates highly dispersed systems.
- Measurable G' and yield point values were obtained at 8 weight percent solids for M-OHWE fine tailings from both Syncrude and Suncor ores, indicating the presence of aggregates. For Syncrude ore treated in the OHWE process, G' and yield point values were first observed at 19 weight percent solids.
- Measured rheological values for air-dried fine tailings were greater than those for pressure filtered fine tailings. The magnitude of the differences between samples from CHWE tests was much greater than those from the corresponding OHWE tests, reflecting the effects of changes in the chemistry of CHWE fine tailings on their rheological properties.
- For CHWE fine tailings from Suncor ore, G' values were about five times higher than those for fine tailings from Syncrude ore. For OHWE fine tailings from Suncor ore, G' values were about two-fold higher and yield points were about one and one-half times higher than those for OHWE fine tailings from Syncrude ore.

Index Tests

- Microscopic observation of floc micro structure (aggregate morphology) shows that flocs in OHWE tailings are larger than those in CHWE mature fine tailings (MFT). This size difference correlates with observed differences in settling rate.
- Changes in water chemistry alter the floc micro structure. OHWE tailings can be converted to a CHWE type by addition of bicarbonate (pH reduction) and *vice versa* by acidification followed by addition of lime.
- PSD of the CHWE and OHWE beach runoff samples showed that the latter material did not segregate significantly during settling while the CHWE samples did.

4.1.4 IERT-NRC Characterization Tests

Aqueous suspensions of ultra-fines (<0.3 μm) were separated from the bulk tailings streams by mechanical agitation, followed by mild centrifugation at 200 g for 1 hour. For separation of ultra-fines from PSV tailings the procedure developed for oil sands ores [6] was used. The crude ultra-fines suspensions were split into two parts: one portion was fractionated by floc size using centrifugation at different speeds; the second portion was separated into hydrophilic and bi-wetted components by emulsion flotation with toluene [7]. The latter approach makes use of the surface characteristic of bi-wetted solids that allows them to stabilize emulsions by forming a cohesive film at an oil-water interface.

In order to determine the primary PSD of the ultra-fines fractions it is necessary to disperse the flocculated solids by replacing the natural pore water with a 0.1 weight percent sodium pyrophosphate solution. This is followed by vigorous mechanical agitation of the suspensions. Dispersed ultra-fine solids are then separated into specific particle size fractions by sequential centrifugation steps at different speeds [8].

4.1.4.1 *Observations*

- Solids in samples from OHWE process streams settled faster than those in the corresponding samples from the CHWE trials. This observation correlates with a greater degree of ultra-fines aggregation owing to the higher Ca^{2+} and lower HCO_3^- content in the former cases.
- In all cases Suncor ore produced faster settling streams than did Syncrude ore.
- For all processes the ultra-fines in the PSV middlings were less aggregated than in other streams. Agitation of PSV middlings, in subsequent process steps, results in floc densification.

- Tailings settling rates were different only until the gelation concentration of the ultra-fines fraction was reached. At the gelation concentration steric hindrance inhibits further settling and from this point on all samples behaved similarly.
- Relative to OHWE, the CHWE process produced slower ultra-fines aggregation, allowing coarser solids to settle independently. Consequently, for both of the ores tested, the OHWE process produced non-segregated sediments from PSV middlings and secondary tailings streams. The CHWE process, on the other hand, produced segregated sediments for the same streams and conditions.
- During the transition from secondary tailings to runoff, coarse hydrophilic solids are selectively removed at the beaches. This results in a relative increase in the amount of bi-wetted solids associated with the runoff. This may increase the ultimate strength and rate of formation of the gel network in the tailings pond. In conjunction with the finer size distribution of any remaining coarse hydrophilic solids, this factor could account for the non-segregating behavior of beach runoff streams exhibited even for the CHWE trials. Also, although the concentration of ultra-fines in each stream remains about the same, the contribution of ultra-fines to the total solids content in the run-off streams is much greater than elsewhere.

4.1.5 Comments

A comparison of the information from the two sets of characterization tests is summarized in Table 4-3. In general the conclusions drawn from the tests were in agreement except in the case of the runoff samples. In the latter case the dif-

TABLE 4-3. Comparison of information derived from macro- and micro-testing of OHWE and CHWE tailings samples.

Tailings property	Observation	
	CANMET-WRL	NRC-IERT
Rate of settling:		
Ore effect	Particles in CHWE tailings were more highly charged, indicating greater dispersion.	Ultra-fines content and degree of dispersability were greater for Syncrude ore than for Suncor ore.
Process effect	Floc micro structure shows that flocs in OHWE tailings are larger than those in the corresponding CHWE samples. These observations correlate with settling data.	OHWE tailings samples settle faster than those from the corresponding CHWE tests until the critical gel concentration is reached. At this point settling rates become the same.
Particle aggregation:		
Rate/degree	Rheological measurements indicate aggregation (as measured by gel strength and yield point) is affected by solids concentration.	Relative to OHWE, the CHWE samples showed slower aggregation.
Segregation		The above observation is reflected in the segregated sediments for PSV and secondary tailings streams for CHWE but not OHWE samples.
Water chemistry	CHWE and OHWE tailings are interconvertible by chemical modification.	Settling behavior is determined by water chemistry differences, particularly Ca^{2+} and HCO_3^- concentration.
Beach run-offs	PSD measurements showed that the OHWE samples exhibited non-segregating behavior while CHWE samples did not.	Bi-wetted solids concentrated in these samples. Samples from both processes showed largely non-segregating behavior.

ferent conclusions may be ascribed to the indefinite nature of the term non-segregating.

Drying and reconstitution of tailings samples is not recommended because the colloidal properties of ultra-fines are irrevocably destroyed during drying and the particles cannot be redispersed. In order to study the effect of water chemistry it is necessary to displace the natural pore water by repeated washings with the replacement liquid. This process has been found to be completely reversible.

4.1.6 References

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4.2 Geotechnical Aspects of OSLO Process Fine Tails

4.2.1 Introduction

The objective of investigations into the geotechnical properties of OSLO Hot Water Extraction (OHWE) and OSLO Cold Water Extraction (OCWE) process fine tailings is to determine the engineering parameters that will control the long term disposal behavior of this material. Compressibility, hydraulic conductivity, and shear strength are the geotechnical parameters that control the long term behavior of fine tails.

The amount of compressibility of fine tails is shown by the decrease in the void ratio with increasing effective stress. This relationship governs how much the fine tails compresses when subjected to ever increasing loads. The increased loading of a mass of fine tails drives water out from within the void space between mineral particles. The hydraulic conductivity of the fine tails governs the rate of this water release, that is, the rate of compressibility. Thus, the variation in hydraulic conductivity with void ratio is also an important relationship. The shear strength of the material will determine what load, such as a sand layer, may be placed on the surface of a mass of fine tails to increase the rate and amount of compression.

Fine tails also exhibit a gain in gel or thixotropic strength. Previous research on fine tails indicates that thixotropic strength continues to increase for over a year. Such increases in strength may significantly reduce the compressibility of the fine tails. All of the above relationships must be quantified in order to determine the long term disposal behavior of fine tails.

Another objective of a major geotechnical research program at the University of Alberta by Miller [1,2] is to compare the behavior of OSLO process fine tails with Clark process fine tails produced during similar pilot plant operations. These pilot plant fine tails will be compared with Clark process fine tails collected from the Suncor and Syncrude tailings ponds. The comparison between pilot plant OSLO and Clark fine tails allows the relative performance of fine tails created under similar conditions to be examined and the reasons governing their behavioural differences to be understood. Behavior differences have been observed, as well, between pilot plant and field fine tails. An understanding of these differences is required to determine the expected long term disposal geotechnical behavior of the OSLO process fine tails produced from a commercial large scale operation.

4.2.1.1 *Description of Extraction Processes*

The current extraction method used by the oil sands industry is the Clark Hot Water Extraction (CHWE) process. In this process, oil sand is mixed with hot water and steam in a tumbler. The resultant slurry is heated to 80°C and a small quantity of sodium hydroxide is added as a process aid. The bitumen is separated from the solids and bulk water through gravity and flotation in a particle separation vessel (PSV).

The OSLO Hot Water Extraction (OHWE) process is also conducted at a temperature of 80°C. Methyl Isobutyl

Carbinol (30 to 125 ppm) and kerosene (60 to 250 ppm) are the process chemicals used. Normally, chemicals are added with the slurry water in the tumbler, but may be added to the flooded slurry box or the feed to secondary flotation. Air is added to the flooded slurry (typically between 0.3 to 0.6 air/slurry volume ratio). Increased air/bitumen attachment time is created by the use of an additional pipe section between the aerator and PSV.

The OSLO Cold Water Extraction (OCWE) process is performed at lower temperatures than the OHWE process. As the conditioning and bitumen separation reactions are only slightly different, the resulting fine tails may have similar geotechnical properties.

4.2.1.2 *Origins and Index Properties of Fine Tails Examined*

The OSLO process fine tails and the comparable CHWE fine tails examined in this paper were obtained at different times from different locations. The origin of each of the fine tails are given in the cited references. The appropriate reports should be referred to for complete descriptions of the fine tails. To ensure that the geotechnical properties of the various fine tails can be compared and to assist with understanding any differences, the following index properties of the fine tails are summarized in Table 4-4: solids content, bitumen content by dry weight (not by the more conventional oil sands total mass basis), and the Atterberg limits (liquid and plastic limits). The amount of material smaller than 44 μm (fines content) and 2 μm (clay size) is summarized in Table 4-4. The grain size distributions from all the references have been replotted at the same scale for comparison purposes.

A testing program was undertaken by Komex Consultants Ltd. [3] that examined both the OHWE and OCWE fine tails. The fine tailings of the OHWE process were modeled using the underflow from a flotation cell routed through a clarifier. These tailings were produced at the OSLO pilot facility from January to March 1990. The grain size distributions of the OHWE and OCWE materials are presented in Figure 4-1. Golder Associates Ltd. [4] tested material from the OHWE clarifier feed from the Petro Canada pilot plant and OCWE fine tails sampled from Fines Pond #2 and fresh OCWE fine tails, both sampled at Lease 41. Grain size distributions for these materials are given in Figure 4-2. Sego [5] investigated the consolidation behavior of never frozen OCWE fine tails as part of a freeze-thaw dewatering research program. The OCWE fine tails were obtained from the fines pond at the Lease 41 site. The grain size distribution of this material is given in Figure 4-3.

A research program is being conducted at the University of Alberta by Miller [2] to compare the geotechnical properties of the fine tails produced from the OHWE and CHWE processes. A cooperative project involving Syncrude, Suncor, OSLO New Ventures, and the Fine Tails Fundamen-

TABLE 4-4. Index properties of OSLO and Clark process fine tails.

Investigator	Year of report	Process	Material	Location	Solids content (%)	Fines content (%)	% less than 2 μm (%)	Bitumen content (%)	Atterberg limits	
									Liquid limit	Plastic limit
Komex [11]	1990	OHWE	clarifier underflow	OSLO pilot	5 to 20	90	48	10 to 20	52	17
Komex [11]	1990	OCWE	fines pond	Lease 41	20 to 40	98	55	5 to 20	45	15
Komex [11]	1990	OCWE	fine tails stream	Lease 41	10 to 20	98	65	5 to 12	60	21
Golder [12]	1990	OCWE	clarifier feed	Calgary pilot	13	95	35	–	68	5
Golder [12]	1990	OCWE	fines pond #2	Lease 41	32 to 37	93 to 99	42 to 51	10	58 to 64	15 to 18
Golder [12]	1990	OCWE	fines pond #1	Lease 41	23 to 27	93 to 99	42 to 51	–	61 to 64	16 to 19
Golder [12]	1990	OCWE	fresh tailings	Lease 41	3 to 4	99	41	2	62	24
Sego [13]	1994	OCWE	fines pond	Lease 41	25	93	49	4	53	24
Miller [9]	1995	OHWE	Suncor treated	Syncrude EEC	22	99	5	6	58	28
Miller [9]	1995	OHWE	Syncrude treated	Syncrude EEC	22	98	15	5	60	31
Miller [9]	1995	OHWE	Syncrude untreated	Syncrude EEC	22	99	47	5	55	28
Miller [9]	1995	CHWE	Suncor ore	Syncrude EEC	22	99	50	6	52	27
Miller [9]	1995	CHWE	Syncrude ore	Syncrude EEC	22	99	45	4	50	26

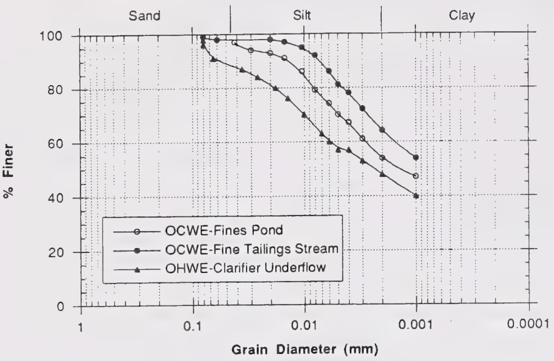

FIGURE 4-1. Grain size distributions of OHWE and OCWE fine tails [11].

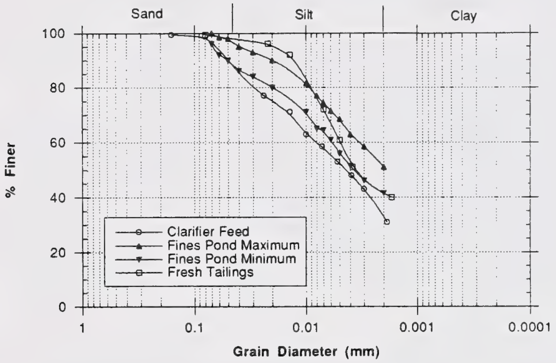

FIGURE 4-2. Grain size distributions of OCWE Suncor fine tails [12].

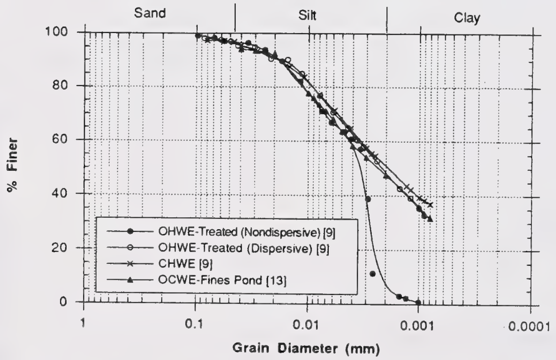

FIGURE 4-3. Grain size distribution of OHWE, OCWE, and CHWE fine tails from Suncor ore.

tals Consortium was initiated and pilot plant extraction runs of the OHWE and CHWE processes were performed using the Syncrude Research Experimental Extraction Circuit (EEC) in October and November of 1993. Five flume tests were performed using combined tailings during which the flume runoff (fine tails) was collected. Details of the flume tests are provided by Miller *et al.* [1] and Lowe *et al.* [6]. This research program had the advantage of using both Syncrude ore and Suncor ore [7] and both extraction processes were performed with each ore for comparison of the resulting fine tails. The process conditions for the five flume tests and the resulting seven fine tails materials being tested are shown in Table 4-5. The index properties measured for these materials are summarized in Table 4-4. A typical particle size distribution of an OHWE and a CHWE process fine tails collected during this research program is presented in Figure 4-3. The results of a comprehensive analysis of the index properties and both nondispersive and dispersive grain size distributions of the combined tailings, beach deposits, and fine tails of all five flume test materials is given by Miller *et al.* [1].

4.2.1.3 *Comparison of Index Properties*

OHWE and OCWE fine tails tested by Komex [3] had high bitumen contents because of upset conditions during extraction. The OCWE fine tails collected from fines pond #2 [4] also had high bitumen contents. No explanation for the high bitumen content of these fine tails was available. All of the other fine tails had typically low bitumen contents of 6 to 7% by dry mass, similar to values seen in Syncrude and Suncor tailings ponds. High bitumen contents have an affect on compressibility and hydraulic conductivity. At high void ratios, compressibility and hydraulic conductivity decrease as bitumen content increases. High bitumen contents, therefore, may have affected the geotechnical properties of the fine tails tested by Komex [3].

Dispersive grain size test procedures completely disperse shale particles into individual soil grains. Shale particles not dispersed in the extraction process are not dispersed in nondispersive particle size test procedures and the actual particle sizes are measured [1]. Dispersed tests do not represent the true grain size of the particles for OSLO materials. Figure 4-3 shows the difference in particle size distribution from a nondispersive test and a dispersive test for an OHWE fine tails. CHWE fine tails have the same grain size distribution as dispersed OHWE fine tails, indicating that CHWE fine tails have already been dispersed in the extraction process. These test results also show that the OHWE and CHWE fine tails are composed of the same amount of clay size fines and ultrafines, the only difference is in the amount

TABLE 4-5. OHWE/CHWE flume test process conditions and fine tails.

Oil sand ore	Process	Water	Fine tails
Syncrude	OHWE	Untreated river	As collected
Syncrude	OHWE	Treated river	As collected
Syncrude	CHWE	Recycled pond	As collected
Suncor	CHWE	Recycled pond	As collected
Suncor	OHWE	Treated river	As collected
Syncrude	CHWE	Recycled pond	600 ppm $CaSO_4$ added
Suncor	CHWE	Recycled pond	600 ppm $CaSO_4$ added

of dispersion of the shale layers which are the source of the fines in the ore and in the tailings stream. Komex, Golder, and Sego's tests all appear to be dispersed and are not truly representative of the OSLO fine tails particle size.

Atterberg limits are indicative of the clay mineralogy, soil chemistry, aqueous phase chemistry, and silt content of fine grained soils and can be correlated with their geotechnical properties. Standard ASTM test methods for Atterberg limits cannot be used with fine tails due to the presence of bitumen. Fine tails should not be bitumen extracted or dried and process water instead of distilled water should be used during Atterberg limits testing. The Atterberg limits for all the fine tails indicate a high plasticity soil but cannot be compared further because the procedures followed by Komex [3] and Golder [4] are not known.

4.2.2 OHWE/CHWE Flume Tests

Flume tests were performed to identify the fines that form the fine tails and determine the extent of hydraulic sorting down the beach, washing of fines out of the beach, and the amount of dispersion of fines in the fine tails investigated. Five flume tests were performed using combined tailings produced during extraction pilot runs of the OHWE and CHWE processes using the Syncrude Research Experimental Extraction Circuit. The flume tests were performed to compare the geotechnical properties of the tailings produced by the OHWE and CHWE processes for both Syncrude ore and Suncor ore.

Miller *et al.* [1] provided a complete discussion of the flume design, the flume test procedures, and the beach and runoff sampling. Beach deposit and flume runoff characteristics were summarized and detailed results of all tests are given. The results of an extensive particle and soil grain size distribution testing program on combined tailings, beach deposit, and flume runoff materials was provided and procedures followed to preserve the chemical and physical characteristics of these materials was also outlined. A comprehensive analysis of the flume tests will be presented as part of a Ph.D. thesis in Geoenvironmental Engineering by Warren G. Miller at the University of Alberta. Comparisons with the results of previous research performed on tailings materials and the application and incorporation of current beach deposition and beach runoff theories will be an essential component of this analysis.

The flume was designed based on Küpper's [8] laboratory flume studies. In this work, several flume deposition tests carried out in different parts of the world to study hydraulic fills are compared and discussed. The results of all test programs are coherent and consistent with field observations of hydraulic fills and natural alluvial deposits, which suggests that, at least qualitatively, flume tests are adequate to simulate the physical phenomena associated with hydraulic deposition. The flume tests of Miller [1] were consistent with this work.

Mean grain size did not change with distance downstream in the flume for both the OHWE and CHWE tailings indicating hydraulic sorting was minimal. Fines not retained in the beach during deposition escape into the fine tails due to segregation mechanisms, not by sorting mechanisms.

Table 4-6 shows that the beach fines and flume runoff fines of the OHWE tailings for both the Syncrude and Suncor ores had approximately the same amount of clay sized (<2 μm) and ultrafine sized (<1 μm) material. However, the CHWE process flume runoff fines had more clay sized and ultrafine material than the beach deposit. Thus, the finer sized fines were washed out of the beach deposit during the deposition of CHWE process tailings, but were not washed out during deposition of the OHWE process tailings.

TABLE 4-6. Fines fraction of beach and flume runoff fines.

Process	Ore	Water	Beach fines		Flume runoff fines		
			< 2 μm (%)	< 1 μm (%)	nondispersive <2 μm (%)	dispersive <2 μm (%)	nondispersive <1 μm (%)
CHWE	Syncrude	Recycled pond	30	22	45	45	18
OHWE	Syncrude	Treated river	15	5	15	40	5
OHWE	Syncrude	Untreated river	25	15	47	43	15
CHWE	Suncor	Recycled pond	33	25	50	50	49
OHWE	Suncor	Treated river	7	1	5	48	1

The nondispersive and dispersive clay size fractions for all five flume runoff fines are also provided in Table 4-6. Using treated river water, the OHWE extraction process results in fines that are not fully dispersed and exist partially as larger shale particles. The OHWE untreated river water and the CHWE extraction processes result in fines that are more fully dispersed and have finer particle size distributions.

4.2.3 Standpipe Tests

Standpipe tests are performed to determine the compressibility and hydraulic conductivity of the fine tails under self weight loading. The relationship between effective stress and void ratio of the fine tails at the end of consolidation, that is, when the excess pore water pressures are dissipated, provides the amount of compressibility at relatively high void ratios. Hydraulic conductivity of the fine tails at the starting void ratio (solids content) may be determined from the initial rate of compression. Manometers are used in standpipe tests to measure excess pore pressures and thus determine when consolidation is complete.

4.2.3.1 *Komex Tests on OHWE and OCWE Fine Tails*

A laboratory program was undertaken by Komex [3] that tested both OHWE and OCWE fine tails. The standpipe tests were performed in 1 L or 0.5 L graduated cylinders. Fine tailings were mixed with either process water or distilled water to create samples with starting solids contents of 5 and 10%. The OCWE fines tailings stream had consistently longer induction periods, but all other measured parameters were somewhat similar between the two materials. The tests were not continued to full consolidation and therefore the final solids contents of these tests were not measured. Compressibility data, therefore, is not available. Detailed results of these tests are provided by Komex [3].

4.2.3.2 *Golder Tests on OCWE Fine Tails*

Standpipe tests were performed by Golder [4] to provide a laboratory simulation of the settlement behavior of the OCWE process fines. The standpipes used by Golder were 2 metres high with a diameter of 102 mm and were equipped with manometers for pore pressure measurements. Golder used City of Calgary tap water when it was necessary to achieve a desired solids content by adding water. Detailed observations and results of this set of standpipe tests is provided by Golder. The void ratio-effective stress data for these standpipe tests is provided in the following Section 4.2.4. The hydraulic conductivity-void ratio relationship is also given in Section 4.2.4.

The standpipe tests performed by Golder resulted in several noteworthy observations. Their results indicated that the boundary conditions in standpipe tests, such as wall friction and arching of the material across the diameter of the standpipe, were not a major concern. As well, an aging effect in the fine tails was observed. Fresh tailings settled at a slower rate than tailings that had been aged in the Lease 41 tailings pond for one year. This conclusion, however, was based on standpipe tests that had significantly different initial solids contents and the variation in test results may be a result of this difference in initial conditions. All slurry materials settle to different solids contents if their initial solids vary. Finally, standpipe tests using bottom drainage resulted in a zone of increased solids content material at the bottom of the standpipe. This blinding layer resulted in decreased hydraulic conductivity and reduced the benefits of the shortened drainage paths created by top and bottom drainage.

4.2.3.3 *Miller Tests on OHWE and CHWE Fine Tails*

Standpipe tests are being performed by Miller [2] at the University of Alberta on the OHWE and CHWE fine tails collected from the pilot plant flume tests conducted at

Syncrude Research. The process conditions for the five flume tests and the resulting seven fine tails materials being tested are shown in Table 4-5. The standpipe tests are being performed to determine compressibility and initial permeability of fine tailings under self weight consolidation. Consolidation has two components: compression and dissipation of excess pore pressures. Excess pore pressure is defined as the difference between the total pore pressure and the hydrostatic pressure. Even when compression of the material appears to have ceased, the consolidation is not complete until all excess pore pressure has been dissipated. The normalized compression and percent consolidation of the two metre, one metre, and half metre high standpipes at an elapsed time are summarized in Table 4-7.

The OHWE extraction process used both untreated river water and treated river water with the Syncrude ore. For simplicity in discussing the different fine tails, when treated river water fine tails are discussed, they are identified as treated fine tails. Similarly, when untreated river water fine tails are discussed, they are identified as untreated fine tails.

Two Metre High Standpipes — Seven two-metre high standpipes containing the seven different fine tails at solids contents of 22% were commenced during May of 1994. The two-metre high standpipes were designed for double drainage. The consolidation of the seven fine tails in the two-metre high standpipes in terms of settlement is presented in Figure 4-4. Examining the Syncrude ore fine tails after 250 days, the CHWE fine tails had settled the same amount as the OHWE untreated fine tails. The CHWE fine tails, however, had settled slightly less than the OHWE treated fine tails. Since the two-metre high standpipes have bottom drainage, the bottom manometer cannot be used to determine percent consolidation. Percent consolidation is defined as the change in pore pressure divided by the initial excess pore pressure. Excess pore pressure profiles provide a better understanding of the consolidation behavior of the fine tails. Figure 4-5 provides the pore pressure profiles of the Syncrude ore samples after 250 days. The OHWE treated fine tails displayed marginally better excess pore pressure dissipation than the CHWE fine tails. The OHWE untreated fine tails had excess pore pressures similar to the CHWE fine tails.

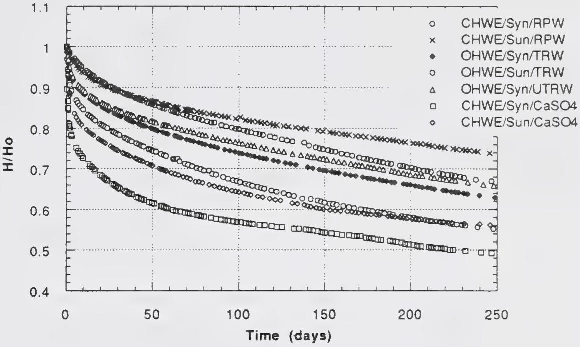

FIGURE 4-4. Consolidation of the fine tails for the 2 m standpipes.

The differences in settlement behavior and dissipation of pore pressure between the OHWE and CHWE process fine tails was more pronounced for the Suncor ore. After 250 days, the OHWE fine tails had settled 19% more than the CHWE fine tails. The excess pore pressure profiles for the Suncor ore in Figure 4-6 clearly shows the superior consolidation behavior of the OHWE fine tails.

TABLE 4-7. Settlement and percent consolidation of two, one, and half metre high OHWE/CHWE standpipes.

Process	Ore	Water	2 metre high (after 250 days)		1 metre high (after 230 days)		0.5 metre high (after 190 days)	
			H/Ho	% consol.	H/Ho	% consol.	H/Ho	% consol.
CHWE	Syncrude	Recycled pond	0.66	31	0.59	61	0.65	50
OHWE	Syncrude	Treated river	0.63	35	0.58	71	0.59	76
OHWE	Syncrude	Untreated river	0.66	33	0.63	41	0.64	52
CHWE	Suncor	Recycled pond	0.74	22	0.74	35	0.80	31
OHWE	Suncor	Treated river	0.55	70	0.58	87	0.60	100

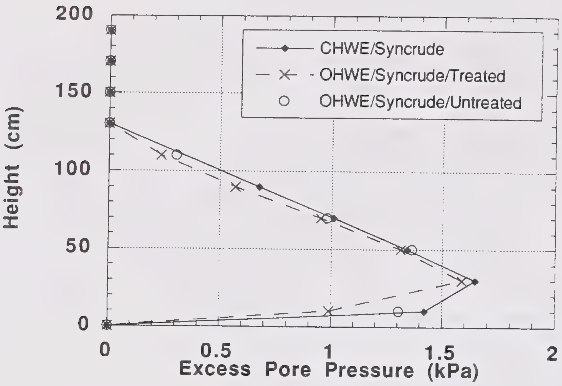

FIGURE 4-5. Two metre high standpipe pore pressure profile after 250 days — Syncrude ore.

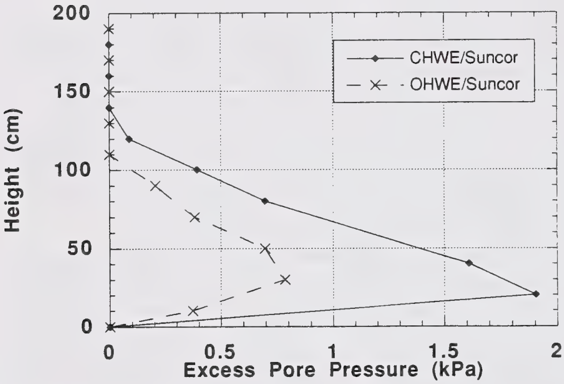

FIGURE 4-6. Two metre high standpipe pore pressure profile after 250 days — Suncor ore.

Previously, an aging effect in the OSLO fine tails examined by Golder [4] was suggested. There appears to be an opposite aging effect visible between pilot plant and field CHWE fine tails. Clark process tailings were sampled from the Syncrude tailings pond and 2-metre high standpipe tests were performed [9]. After 250 days, pilot plant CHWE fine tails had settled approximately 29% more than the field CHWE fine tails. These results point out that compressibility and hydraulic conductivity relationships generated from tests on new pilot plant fine tails may only be used to identify behavior trends of the fine tails and that long-term laboratory or field tests are necessary to accurately define fine tails behavior for commercial plant tailings ponds.

After approximately 200 days, the solids contents with depth in the seven 2-metre high standpipes were measured [2]. All the fine tails exhibited the same general trend except the $CaSO_4$ treated fine tails. Similar to Golder's [4] results for standpipes with both top and bottom drainage, the solids content was consistent along the height of the standpipe then began to increase rapidly close to the base of the standpipe. This increase in solids content is due to bottom drainage and results in decreased hydraulic conductivity that reduces the effectiveness of bottom drainage of the fine tails. However, the CHWE fine tails with $CaSO_4$ showed a slightly different trend with a consistent increase in solids content with depth. A complete discussion of these results is included in Miller *et al.* [2].

One Metre High Standpipes — During June of 1994, 1-metre high standpipe tests with top drainage only were commenced using the same materials and initial solids content of 22% used in the 2-metre high standpipes. Table 4-7 summarizes the settlement and percent consolidation of the 1-metre high standpipes after 230 days. Results were similar to the 2-metre high standpipes. With Syncrude ore, OHWE treated fine tails displayed marginally better geotechnical behavior than the OHWE untreated and CHWE fine tails. The initial settling rate of both OHWE fine tails during the first 100 days was greater than the CHWE fine tails. For the Suncor ore, the OHWE fine tails had settled considerably more than the CHWE. The OHWE fine tails had also reached a percent consolidation of 87%, compared to only 35% for the CHWE fine tails.

Half Metre High Standpipes — An accelerated standpipe testing program was also performed using 0.5-metre high standpipes. This testing program has been completed and the results from the self weight consolidation phase and the variation of solids content with depth have been reported [10]. This information was used to determine the compressibility relationships for the different materials at relatively high void ratios. Tests were conducted using double drainage at initial solids contents of 22% and 30% and single drainage at 22% initial solids content. Complete results of these tests are provided by Mazurek *et al.* [10].

Table 4-7 summarizes the settlement and percent consolidation of the top drainage only 22% solids standpipes at the end of testing (approximately 190 days). The improved geotechnical behavior trends discussed previously in the two and one metre high standpipe tests were observed again in the 0.5-metre standpipe test results. OHWE treated fine tails from Syncrude ore were only marginally better than comparable CHWE fine tails and Suncor ore OHWE fine tails were considerably better than their CHWE counterparts. The compressibility relationships derived from these tests will be discussed in Section 4.2.4.2.

Miller's standpipe test results on OHWE and CHWE fine tails have indicated significant geotechnical trends in the rate of compressibility and pore pressure dissipation. OHWE fine tails from Suncor ore showed significantly better geotechnical behavior than comparable fine tails from the CHWE process. OHWE fine tails from Syncrude ore showed a marginal improvement in behavior over the fine tails from the CHWE process. Thus, whether the geotechnical engineering properties of OHWE fine tails are better than those of CHWE fine tails appears to be dependent on the chemical and physical properties of the ore and the chemistry of the process water.

4.2.4 Compressibility/Hydraulic Conductivity Testing

Consolidation/hydraulic conductivity tests are performed to determine the amount and rate of compressibility over a wide range of effective stresses and to determine the hydraulic conductivity over a range of void ratios. These relationships may be combined with comparable relationships obtained from standpipe tests performed at higher void ratios to provide the compressibility and hydraulic conductivity of the fine tails over a complete range of void ratios. These results are used in finite strain consolidation models to determine the long term disposal behavior of the fine tails in the field.

4.2.4.1 *Testing Programs*

Consolidation tests were performed by Komex [3] on the OHWE fine tails and OCWE fine tailings stream. A constant rate of strain consolidation test was used. The fine tails were first dried to a point where they would not display segregation characteristics. Such drying of the fine tails will result in a concentration of process water chemicals which may have an effect on the geotechnical behavior of the fine tails. The compressibility relationships of these OHWE and OCWE fine tails are shown in Figure 4-7. No measurements of hydraulic conductivity were reported for these tests.

Golder [4] performed consolidation tests on OCWE fine tails produced from Suncor ore at Lease 41 at solids contents greater than 30%. That is, at a point when effective stresses were thought to exist in the fine tails. Documented evidence that hydraulic conductivity of oil sand fines was strongly dependent on hydraulic gradient resulted in Golder using three types of consolidation tests: conventional incremental load, constant rate of deformation, and controlled gradient. Figure 4-7 also provides the compressibility relationship for the OCWE fine tails tested by Golder. Hydraulic conductivity was estimated from the compressibility-time measurements. Figure 4-8 presents their relationship between hydraulic conductivity and void ratio.

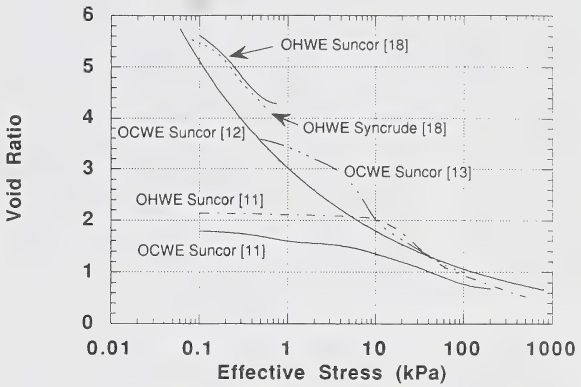

FIGURE 4-7. Compressibility of Suncor OHWE, Syncrude OHWE, and Suncor OCWE fine tails.

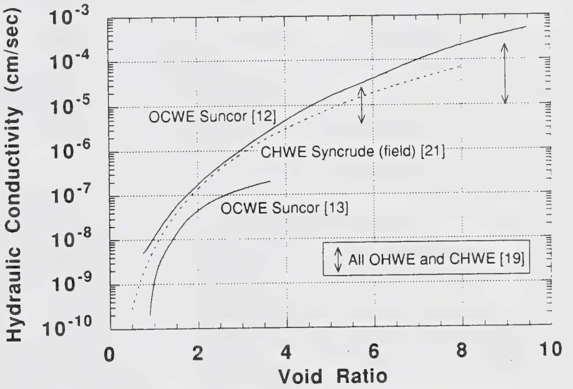

FIGURE 4-8. Hydraulic conductivity of OSLO and Clark fine tails.

Sego [5] used incremental loading consolidation tests to measure compressibility and hydraulic conductivity of the OCWE fine tails from the same Lease 41 tailings pond. Figure 4-7 also shows the compressibility of these OCWE fine tails. These results were reported to be typical of high moisture content fine mine wastes. As the fine tails are loaded and the void ratio decreased, there is a dramatic decrease in hydraulic conductivity. As shown in Figure 4-8, a decrease in void ratio from 5 to 1 causes a decrease in hydraulic conductivity of 3 orders of magnitude.

Consolidation tests are currently being performed by Miller [2] on OHWE and CHWE pilot plant fine tails. Incremental loading consolidation tests are being used. The compressibility and hydraulic conductivity as a function of void ratio will be determined and combined with comparable relationships derived from the two, one and half metre high standpipe tests. There are currently 14 consolidation/hydraulic conductivity/shear strength tests underway started at initial solids contents of 22%. One double drainage and one single drainage test are being performed on each of the seven fine tails.

Compressibility and hydraulic conductivity results from the half metre high standpipe tests are given in Figures 4-7 and 4-8, respectively. Complete results of all half metre high standpipe test compressibility measurements are given by Mazurek *et al.* [10] and complete results of measurements of hydraulic conductivity are also provided [11].

4.2.4.2 *Summary of Compressibility and Hydraulic Conductivity Test Results*

The wide range of results in Figure 4-7 shows the importance of test procedure in determining the relationship between void ratio and effective stress. Void ratios at low effective stresses (less than 3 kPa) are determined from standpipe tests which take a long time to fully consolidate. If pore pressures are not measured by manometers, the degree of consolidation is not known. As there are no standardized procedures for such standpipe tests, experimental results from different laboratories often cannot be compared.

Figure 4-7 shows the amount of compressibility when the fine tails are fully consolidated. That is, no excess pore pressures exist. The time to reach full consolidation, or the rate of compressibility is not a factor in this relationship. The rate of compression is determined by the hydraulic conductivity and amount of compressibility of the material. As an example, the standpipe data has shown that Suncor OHWE fine tails consolidate considerably faster than the Syncrude OHWE fine tails. Figure 4-7, however, shows that the Syncrude OHWE fine tails will eventually undergo a slightly greater amount of compression than the Suncor OHWE fine tails. Fine tails in the field may never achieve the low void ratios shown in this figure in a reasonable length of time. All the relationships shown in Figure 4-7, therefore, have to be considered with the realization that the time to reach this condition is not taken into account.

At low effective stress, the soil chemistry and aqueous phase chemistry play an important role in the resulting void ratio of the fine tails. At higher effective stresses (greater than 10 kPa), the applied stress dominates the behavior of the fine tails. All the materials in Figure 4-7 have similar void ratio-effective stress relationships over 10 kPa and they do not differ greatly from CHWE fine tails from the Syncrude tailings pond [12]. Komex [3] results at effective stresses less than 10 kPa do not fit into the rest of the data, and they appear to be a function of the test procedure used. Constant rate of strain tests on slurry materials result in a blinding layer forming in the apparatus and excessively high pore pressures being measured. It also appears that they initially dried the fine tails to high solids contents but the detailed test procedures are not given.

To determine hydraulic conductivity at high void ratios, standpipe tests have to be used. Hydraulic conductivity is calculated by assuming the material in the standpipe undergoes hindered sedimentation during the initial stages of settlement [11]. During this time, no effective stress has been developed and the solid particles settle at a constant void ratio. Most consolidation apparatus are not capable of measuring hydraulic conductivity at high void ratios. However, hydraulic conductivity at lower void ratios can be determined from consolidation tests either directly or indirectly. Direct measurements of hydraulic conductivity are usually made using a constant head permeability test. Indirect measurements can be estimated from the compressibility and coefficient of consolidation. Direct measurements of hydraulic conductivity are considered better than the indirect methods because assumptions used in the indirect calculation are not completely applicable to large strain materials such as fine tails.

Examining Golder's [4] results in Figure 4-8, hydraulic conductivity values at void ratios above 1.6 are based on hindered sedimentation analysis of standpipe test results. Their hydraulic conductivities at void ratios less than 1.6 were calculated from indirect measurements. Sego [5] measured hydraulic conductivities directly but his test procedure did not include measurements above a void ratio of 3. The hydraulic conductivities of Miller [11] and the CHWE fine tails from the Syncrude tailings pond [13] were measured directly.

The CHWE fine tails from the Syncrude tailings pond are shown in Figure 4-8 for comparison. The hydraulic conductivity relationships of the two OCWE fine tails from the fines pond at Lease 41 may differ because of different test procedures. Miller's initial results for all seven OHWE and CHWE fine tails are shown for void ratios of 5.7 and 9.0 and indicate that all these materials have hydraulic conductivities in the range of the CHWE Syncrude tailings pond fine tails. These hydraulic conductivities were calculated from 0.5-metre high standpipe measurements. The 2-metre and 1-metre high standpipe tests, however, indicate hydraulic conductivities as much as an order of magnitude higher and similar to the values measured by Golder. Miller's results also show that within the indicated ranges, the CHWE $CaSO_4$ fine tails appear to have a higher hydraulic conductivity than the OHWE fine tails, which in turn have a higher hydraulic conductivity than the CHWE fine tails.

4.2.5 Undrained Shear Strength of Fine Tails

The shear strength of fine tails depends on the solids content and on time. With time there is a gain in thixotropic strength which depends on the initial solids content, clay mineralogy, soil chemistry, aqueous phase chemistry, and silt content. The undrained shear strength is an important parameter because it will determine the load the fine tails are capable of carrying. The maximum or peak shear strength of fine tails and its increase with time can only be measured with specialized equipment and generally most laboratories only report residual or remoulded shear strengths.

For highly thixotropic materials, such as fine tails, the remoulded shear strength is only a small percentage of the peak shear strength. The thickness of sand layers that may be placed on the fine tails will depend on its peak strength if uniform layers are deposited. Remoulded strength is only a characteristic of remoulded fine tails and is not the correct parameter to determine the load carrying capacity of fine tails deposits unless they are significantly disturbed during sand layer placing.

4.2.5.1 *Remoulded Shear Strength Tests*

Komex [3] measured the remoulded shear strength of the OHWE and OCWE fine tails over a range of solids content using a viscometer. Golder [4] used a similar technique to measure remoulded shear strength of OCWE fines tails derived from Suncor ore. Banas [14] measured the remoulded strength of CHWE fine tails collected from the Syncrude tailings pond using a specially designed vane shear apparatus. Miller [2] used the same apparatus to measure remoulded shear strengths on five of his OHWE and CHWE fine tails materials. The results of all of these tests are provided in Figure 4-9. The OHWE and OCWE fine tails have remoulded undrained shear strengths similar to those of the CHWE fine tails from the Syncrude tailings pond. The most important observation from Figure 4-9 is that the shear strength of all of these materials for solids contents below 40% is less than 0.2 kPa which is not a significant strength.

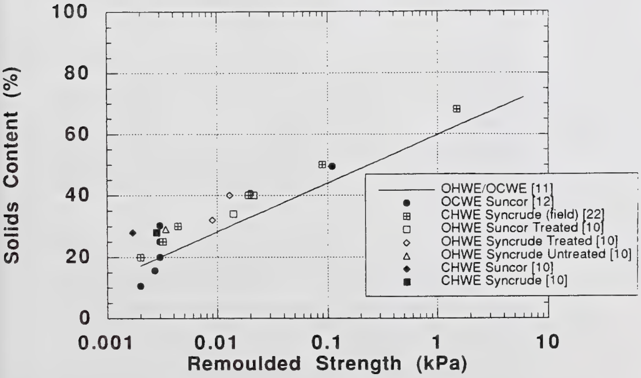

FIGURE 4-9. Remoulded strength of OHWE, OCWE, and CHWE fine tails.

4.2.5.2 *Gain In Thixotropic Undrained Shear Strength*

Miller [2] has measured the gain in thixotropic strength of five of the OHWE and CHWE pilot plant fine tails over a period of 80 days using cavity expansion techniques. The fine tails were tested at a solids content that the fine tails would reach after 365 days of self-weight consolidation. These predicted solids contents were based on consolidation data from 2-metre high standpipes. This approach was used because it would allow the strength of the different fine tails to be compared at the solids content they would have one year after deposition. The gain in peak strength of OHWE and CHWE fine tails derived from Suncor ore is shown in Figure 4-10.

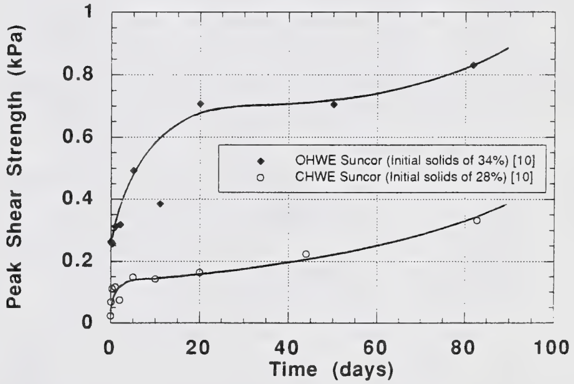

FIGURE 4-10. Peak undrained shear strength of OHWE and CHWE fine tails.

Based on experience with placing fills on muskeg and soft clay deposits, a peak fine tails shear strength of 1 kPa would be required to support a 0.5 metre submerged sand layer. After only 20 days of thixotropic strength gain, Suncor ore OHWE fine tails had a shear strength approaching the value needed to support such a sand layer, whereas the CHWE fine tails had a shear strength less than 0.4 kPa even after 80 days. The OHWE treated fine tails from Syncrude ore required 80 days to approach the required strength. The CHWE and OHWE untreated fine tails from Syncrude ore also had insufficient shear strength after 80 days. These trends indicate that the thixotropic strength is dependent on the chemical and physical characteristics of the ore and on the pore water chemistry.

Currently, another set of thixotropic strength tests are being performed by Miller [2] on the OHWE and CHWE pilot plant fine tails at starting solids contents of 40%. These tests results will be combined with the previous tests at lower solids contents to allow the strength of the fine tails at increasing times and solids contents to be determined.

4.2.6 Overall Summary of Test Results

The objective of the investigation into the geotechnical properties of OSLO process and Clark process fine tails is to determine the engineering parameters that will control the long term disposal behavior of these materials. The deposition characteristics, dispersion of fines, compressibility, hydraulic conductivity, and shear strength of these materials must be considered in determining the geotechnical advantages of the different extraction processes. Investigation of these parameters for various OSLO and Clark process fine tails has led to the following observations.

1. Pilot plant flume tests indicated that during deposition, hydraulic sorting down the beach slope was minimal for both OHWE and CHWE tailings. Thus, fines not retained in the beach escape into the fine tails due to segregation mechanisms, not by sorting mechanisms.
2. Washing of fines out of the beach deposit was investigated based on the $<2\ \mu$m and $<1\ \mu$m fractions in the beach fines and flume runoff fines. These finer sized fines were not washed out of the OHWE beach deposits but were washed out of the CHWE beach deposits.
3. Using treated river water, the OHWE extraction process results in fines that are not fully dispersed and exist partially as larger shale particles. The OHWE untreated river water and the CHWE extraction processes result in fines that were more fully dispersed and have finer particle size distributions.
4. The settlement and consolidation behavior of OHWE and CHWE fine tails were compared by performing standpipe tests with various heights by Miller [2,10,11]. For the Syncrude ore, the OHWE untreated fine tails were similar to the CHWE fine tails with the OHWE treated fine tails only marginally better. However, for the Suncor ore, the settlement and consolidation behavior of the OHWE treated fine tails was considerably better than the CHWE fine tails.
5. At low effective stress, the soil chemistry and aqueous phase chemistry play an important role in the resulting void ratio of the fine tails. However, all OSLO process fine tails have similar compressibility relationships over 10 kPa and they do not differ greatly from CHWE fine tails from the Syncrude tailings pond. These test results which indicate that at higher effective stresses the applied stress not the chemistry dominates the behavior of the fine tails.
6. Fine tails from the OSLO process have hydraulic conductivities in the range of the CHWE Syncrude tailings pond fine tails. Differences between test results of OSLO fine tails may be due to differing test procedures. However, preliminary analysis of Miller's tests [11] shows CHWE $CaSO_4$ fine tails appear to have a higher hydraulic conductivity than the OHWE fine tails, which in turn have a higher hydraulic conductivity than the CHWE fine tails.
7. Based on experience with placing fills on muskeg and soft clay deposits, a peak fine tails shear strength of 1 kPa would be required to support a 0.5 metre submerged sand layer. OHWE fine tails from Suncor ore and Syncrude ore had a shear strength approaching this value after 20 and 80 days, respectively. OHWE untreated fine tails and CHWE fine tails would not be able to support the sand layer even after 80 days. These trends indicate that the thixotropic strength appears to be dependent on the chemical and physical characteristics of the ore and on the pore water chemistry.

4.2.7 Conclusions

The following geotechnical trends in the amount and rate of compressibility and in the rate of pore pressure dissipation have been compared for the OSLO and Clark process fine tails.

- OSLO process fine tails from Suncor ore showed significantly better geotechnical behavior than comparable fine tails from the Clark process. OSLO process fine tails from Syncrude ore, however, had only marginally better geotechnical properties than the Clark process fine tails.
- Whether geotechnical engineering properties of OSLO fine tails are better than those of Clark fine tails appears to be dependent on the chemical and physical properties of the ore and the chemistry of the process water.
- An aging effect in fine tails has been noted for the rate of compressibility between pilot plant fine tails and tailings pond fine tails. Compressibility relationships from tests

on new pilot plant fine tails should only be used at the present time to identify behavior trends of the fine tails. Long term laboratory or field tests are suggested to define fine tails behavior for commercial tailings ponds. Further investigation is required to quantify this aging effect.

4.2.8 References

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5.0 CONCEPTS FOR COMMERCIAL-SCALE CONTAINMENT OF TAILINGS SOLIDS FROM OSLO EXTRACTION PROCESSES AND THEIR LONG-TERM IMPACT ON THE ENVIRONMENT

5.1 Introduction

The OSLO Cold Water and OSLO Hot Water Extraction processes (OCWE and OHWE) generate tailings with similar chemical and physical characteristics. Comparative studies at various scales have shown these tailings to be different from tails generated by the current commercial process. The differences are such that OSLO tailings can be managed either in the same way tailings from the commercial process are managed or in ways that take advantage of their unique properties. An extensive test program was undertaken by the Fine Tails Consortium (FTC) to compare characteristics of OHWE and Clark hot water tailings generated from the same oil sands. Results are reported in Chapter 3.0 of this volume. This paper draws on those results as well as on prior OSLO studies to address disposal options that are potentially available for managing tailings solids from the OSLO processes and the environmental implications of such options.

5.2 Background

Both Syncrude and Suncor use the caustic-based Clark Hot Water Extraction (CHWE) process to recover bitumen from oil sands. In addition to a bituminous froth product, this process generates two tailings streams — one containing coarse solids and another containing fine solids. On leaving the extraction plant, these streams are combined and pumped to a tailings pond or sand storage area in the form of a slurry containing 40 to 55 weight percent solids. Tailings solids segregate during deposition, with the coarse sand fraction settling first followed by the silt fraction. Most of the finer, clay size particles are carried by the runoff water into the settling basin.

Currently, tailings solids are used either to construct (by the cell method) perimeter dykes for the settling basin or to form a large sand dump from which runoff is returned to a separate settling basin. In the settling basin, runoff densifies over three to five years to mature fine tails (MFT) with 25 to 30 weight percent solids. Further dewatering occurs exceedingly slowly, hence the accumulation of MFT. It is believed that clay particles ($<2\ \mu m$) in fine tails have the ability to retain water in an amount that is several orders of magnitude times their own weight, which accounts for the stability of MFT.

Syncrude Canada Ltd. is proposing to place a five-metre deep layer of clean water on top of the MFT to ensure the isolation of the fluid as well as to establish a viable, self-sustaining lake system. Volume II contains further details on this wet landscape reclamation technique. Although Syncrude has carried out pilot tests on the method and although the technology is progressing towards commercial-scale demonstration, there is still a need for alternate techniques that result in reduced landscape disturbance during mining operations and that do not require the storage of MFT in fluid form.

5.3 Modified Wet Landscape Reclamation

In comparison with the CHWE process, tailings generated by the OSLO processes exhibit less dispersion of clays, more rapid initial settling (dewatering) of fine solids, and increased inter-particle strength of the resulting sediment.

Figures 5-1 to 5-6 illustrate the non-dispersive nature of the OHWE process and the fact that it generates less ultra-fine ($<0.2\ \mu m$) material than does the CHWE process. Since it is the dispersed ultra-fines that effectively prevent solids $<2\ \mu m$ (but $>0.2\ \mu m$) from settling in the CHWE process, dyke building and beaching operations in an OHWE operation should result in a greater portion of the fines fraction being trapped in sand voids. Provided adequate permeability is maintained, this type of fines packing should substantially reduce ponding and fines containment areas.

A study carried out at the University of Alberta [previous Chapter] indicated that OHWE fines with their larger-sized particles do exhibit higher permeability than CHWE fines. Both larger-sized particles and higher permeability would allow for improved consolidation of OHWE fine tailings, and the consolidation might be accelerated even further depending on the process used for depositing total tailings. To date all test work has been on the laboratory or small, field pilot scale and the results need to be confirmed on a larger scale.

OSLO fine tailings, because they dewater more rapidly, would require a smaller ponded surface area for clarifying process water before it is recycled. The fastest settling rate for OHWE tailings was obtained by pretreating process water from the Athabasca River to either maintain or reduce the concentration of naturally occurring dispersing agents. Figures 5-7 and 5-8 show initial and long term settling rates for OHWE fine tailings generated from treated river water.

The initial settling rate is several orders of magnitude higher than it is for CHWE tails. However, the long term (≥ 2 years) settled volume does not seem to vary much between the two processes. Thus both processes could produce mature fine tails of similar density (25 to 35 percent solids), with potentially a smaller volume of MFT for the OHWE process. When froth treatment tailings and extraction tailings were combined the settling behavior (Figure 5-8) of OHWE fine tailings was not noticeably altered. This observation is taken as confirmation of the existence of larger-sized particles.

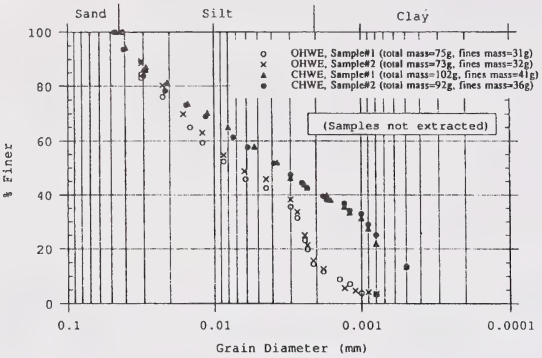


FIGURE 5-1. PSDs (non-dispersed) for OHWE (treated water) and CHWE combined tailings fines (Syncrude oil sand).

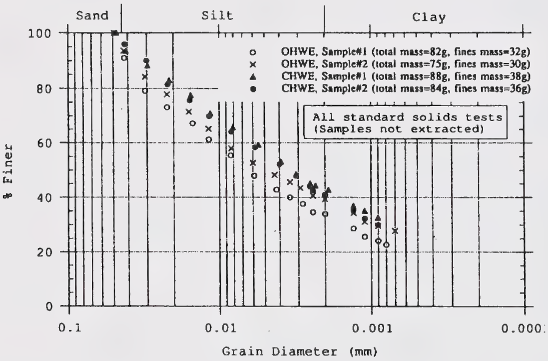


FIGURE 5-2. PSDs (dispersed) of OHWE (treated water) and CHWE combined tailings fines (Syncrude oil sand).

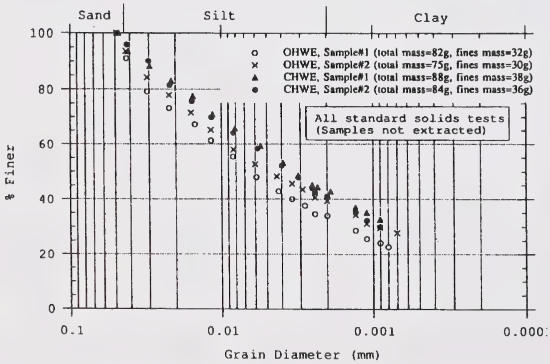


FIGURE 5-3. PSDs (non-dispersed) of OHWE (treated water) and CHWE beach fines (Syncrude oil sand).

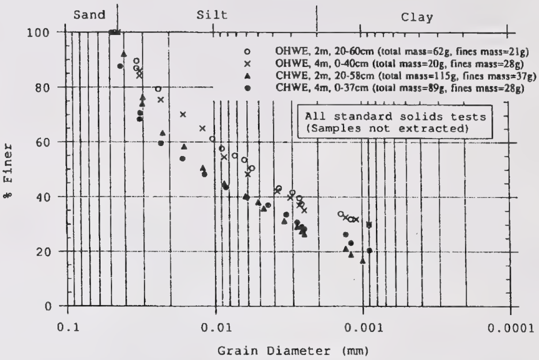


FIGURE 5-4. PSDs (dispersed) of OHWE (treated water) and CHWE beach fines 5 (Syncrude oil sand).

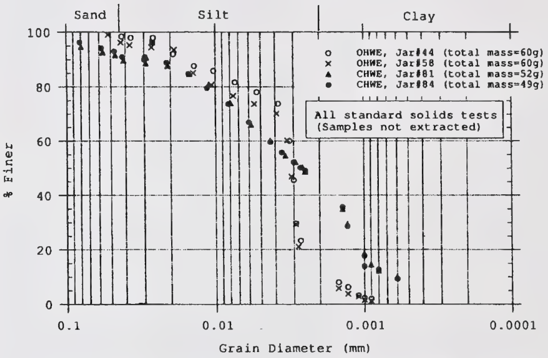


FIGURE 5-5. PSDs (non-dispersed) for OHWE (treated water) and CHWE runoff fines (Syncrude oil sand).

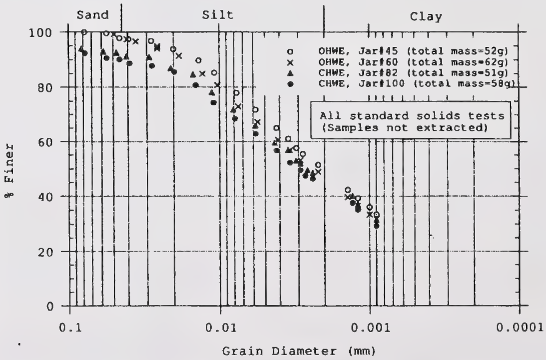


FIGURE 5-6. PSDs (dispersed) for OHWE (treated water) and CHWE runoff fines (Syncrude oil sand).

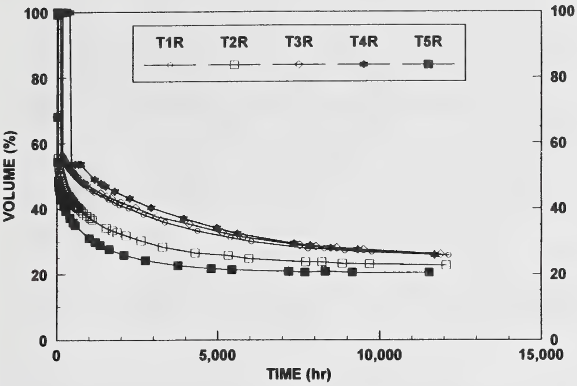


FIGURE 5-7. Fine tails volume *versus* time (T2R = OHWE, treated H_2O, SCL ore).

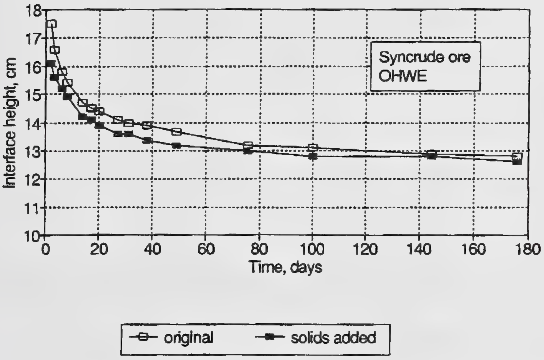


FIGURE 5-8. Settled height of OHWE (treated water) fine tails with and without froth solids added (Syncrude oil sand).

Although the inter-particle strength of sediment is generally low for all processed fines, MFT from the OSLO processes has higher rheological properties than MFT from the CHWE process. In fact the elastic modulus and yield point of OSLO MFT, at 30 to 40 weight percent solids, are three to four times (Figures 5-9 and 5-10) that of CHWE MFT. These enhanced rheological properties may allow for increased entrapment of fines as sand-beach overlaps the MFT contained in the settling basin. It may also allow the method of construction of the settling basin to be revised so as to minimize dyke width.

The current practice, then, of beaching and containment of MFT when applied (with appropriate modifications) to tailings from the OSLO processes may produce the following benefits:

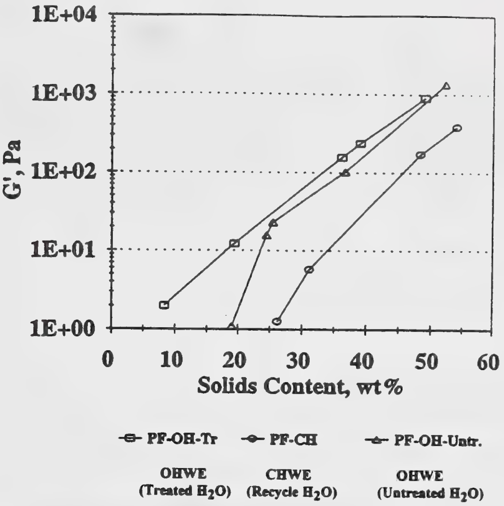


FIGURE 5-9. Elastic modulus *versus* solids content for OHWE and CHWE fine tails (Syncrude ore).

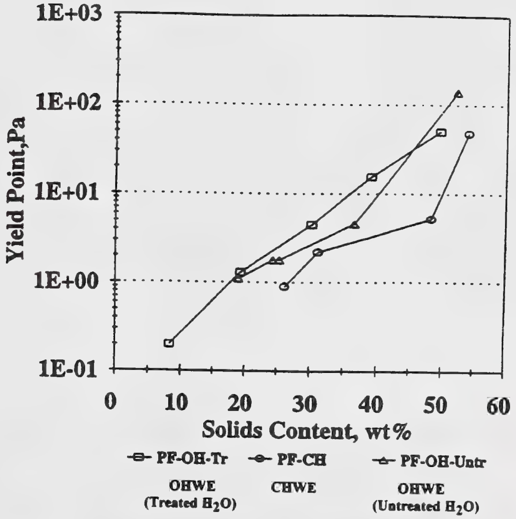


FIGURE 5-10. Yield point *versus* solids content for OHWE and CHWE fine tails (Syncrude oil sand).

- less surface disturbance due to increased entrainment of fines in sand,
- a sand-fines mix with high permeability resulting in a consolidated deposit with high overall strength, and
- a smaller overall settling basin area as compared to the current operations.

5.4 Alternate Disposal Techniques for OSLO Tailings

The unique characteristics of OSLO tailings can also enable the application of disposal techniques such as alternate layering of fines and sand and consolidation of fines by alternately freezing and thawing them. Both techniques were successfully evaluated by OSLO during a field-scale pilot test.

5.4.1 Sand Layering

The layering reclamation concept which consists of alternately layering sand and MFT up to the desired elevation relies on the surface strength of the MFT to support a surcharge of sand. Once an initial sand layer is established, consolidation of MFT would occur over a few years and the contained volumes would decrease by 50 percent as shown in Figure 5-11. Successful layering on the scale of the Syncrude and Suncor tailings operations requires a MFT of about 30 weight percent solids. This method produces a bedded type of deposit with fines sandwiched between sand layers.

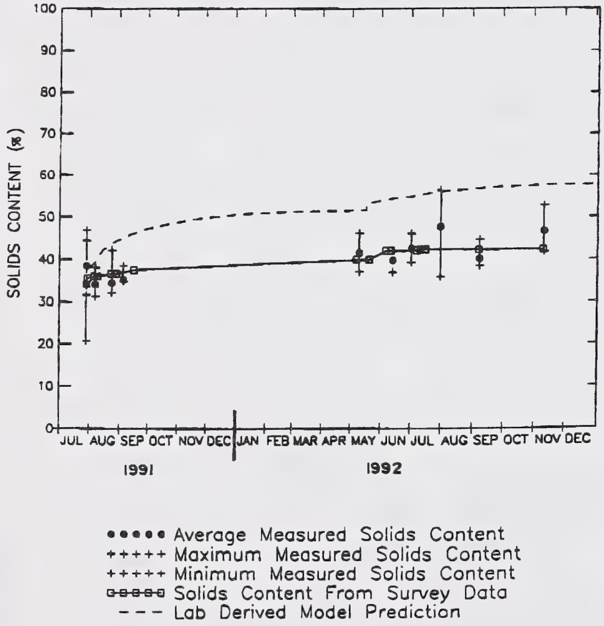


FIGURE 5-11. Solids content of settled fines layer in fines pond #2.

A key enabling factor in sandwiching fines between layers of sand is the initial uniform placement of sand at a rate that does not disturb the MFT surface. If the initial placement is too rapid or uneven, the whole mass of settled fines may overturn as occurred during one of the OSLO field tests and as is reported in the work done by a Florida phosphate mine. Later in the cycle the raining of sand through water might be feasible because, once established, the layering process is self-healing. Sand layering may also be achieved naturally in a settling pond where the sand beach advances out over the contained MFT thus entrapping and locally consolidating the settled fines. The sand layer enables drainage with time and promotes further consolidation for both natural and intentionally created layered systems, as shown in Figure 5-12

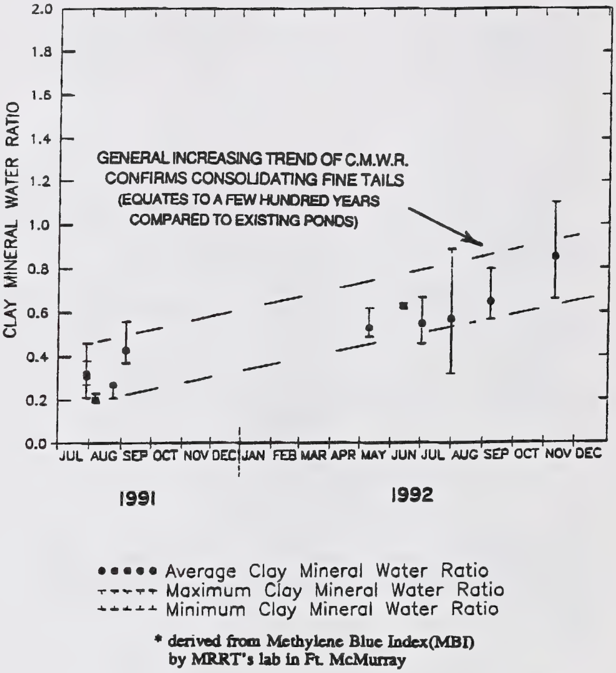


FIGURE 5-12. Clay ratio of settled fines layer in fines pond #2.

In a commercial layering operation, three or four layering ponds would be required to operate both sequentially and in a continuous manner. The pond area would be sized to enable fines to settle to a slurry density of 25 to 30 weight percent solids in a relatively short time period (4 to 6 weeks). The operating sequence proposed by OSLO involves a pond in which fines are settled to a desired consistency, a second pond in which settled fines are undergoing sand loading, and a third pond in which MFT consolidates following sand loading. Land disturbance during the life of the project will be higher than with the proposed modified beaching technique described earlier. However, the layering concept would result in completion of reclamation (with no potential residual liability) sooner than for the beaching case. The layering technique, then, is distinctly different and cannot be combined with the beaching technique.

5.4.2 Freeze-Thaw

Features of the freeze-thaw technique such as rapid volume reduction, post-thaw improvement in the consolidation behavior of fine tails after a single freeze-thaw cycle, and the relatively low toxicity of melt water can be utilized to achieve a reclaimable solids disposal option for the tailings generated in OSLO processes. The climatic conditions in Fort McMurray promote the freezing of fines which upon thawing results in a major reduction (50 percent) in fine tailings volume. This was verified in laboratory and field-scale pilot tests with OSLO tailings. The thawed fines exhibited improved permeability which was caused by clay particles forming peds during the freezing process and maintaining that structure during thaw. The permeability increase is an order to magnitude greater than what was obtained for CHWE process fines, as shown by the hydraulic conductivity plots in Figures 5-13 and 5-14. The data in these figures were generated by testing the OSLO process using oil sands from the Syncrude and Suncor leases.

A commercial operation using the OSLO process would require two or three freeze-thaw ponds for the mature fine tailings. Fine tails of about 25 weight percent solids would be placed in a thin layer on a previously frozen surface layer. The thin layers are alternatively placed and allowed to freeze until the required thickness of frozen mass is achieved. The overall thickness would be dictated by both the thawing rate and the post-thaw dewatering rate which could be enhanced by applying a sand surcharge. Thus every winter subsequent layering of MFT slurry would further consolidate the layers below and four or five cycles would be needed to generate a stable, trafficable deposit. This disposal concept can be practiced either independently or in combination with the sand layering technique.

5.5 Environmental Aspects of Process Water and Leachate

Any of the fines consolidation techniques described earlier can result in the discharge of process-affected water to the environment as a direct or indirect result of:

- release of pore pressure during consolidation,
- drainage or runoff from reclaimed areas,
- ground water contamination.

Volume II describes the technical challenges associated with managing process-affected water for a CHWE operation. OSLO has also generated corresponding test data for it's processes. A recent study sponsored by the Fines Tailings Consortium (FTC) indicates that the long-term toxicity of process-affected water is determined largely by naturally occurring elements in the oil sand itself. Additional toxicity, if generated, is attributable to the types of chemicals used in the process.

Compared with the CHWE process, the OCWE/OHWE processes appear to mobilize lower concentrations of toxic components (in particular naphthenic acids) in process-affected water. Acute toxicity test data showed that OHWE process water was five to seven times less toxic than CHWE

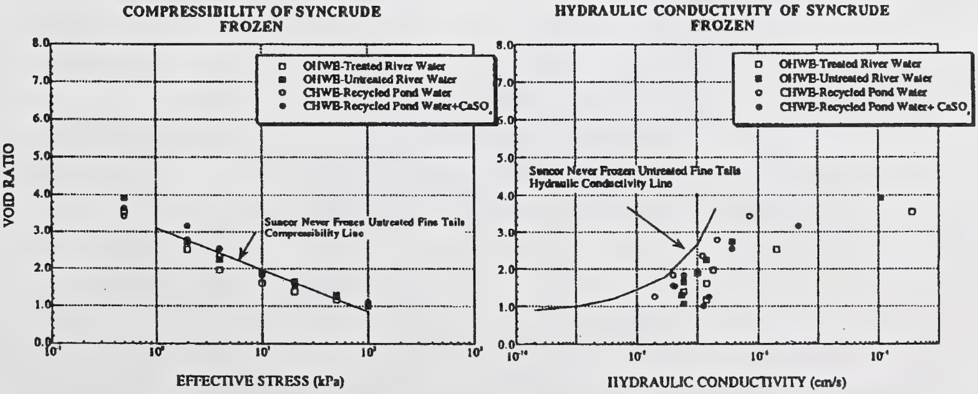


FIGURE 5-13. Comparison of post-thaw consolidation of OHWE (treated H_2O) and CHWE fine tails (Syncrude oil sand).

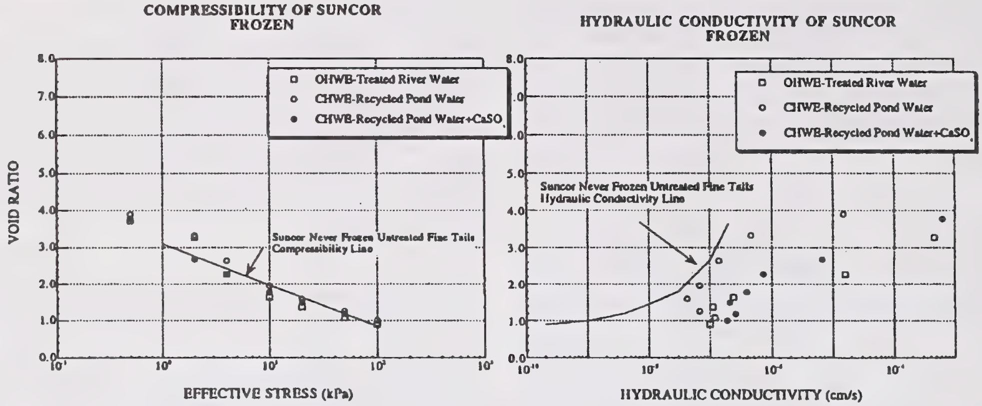


FIGURE 5-14. Comparison of post-thaw consolidation of OHWE (treated H_2O) and CHWE fine tails (Suncor oil sand).

process water and was also deemed “not acutely toxic” after a period of only three months. By comparison, the CHWE process requires a few years to reach a similar status. These comparisons are based on the use of similar oil sands with each process. The conclusion is that process water of a quality suitable for discharge to the environment can be attained in a short period of time (months) if the approach used in managing OSLO process tailings includes the freeze-thaw technique.

Leachate studies done in 1990 by Golder Associates (Calgary) on OCWE tailings materials from the Lease 41 pilot test site categorized both the fine tails and the beach solids as good subsurface materials for reclamation purposes. The 1990 study by Monenco drew the same conclusions. Biological toxicity analysis (Microtox) of extracts from beach sand and fine tails samples showed no acute toxicity. Fish bioassay tests (96-hour basis) also showed no mortality although some test subjects were visibly stressed in extracts of the fines tails samples.

Golder Associates carried out further leachate studies in 1992 using samples of settled fines that were undergoing consolidation on the Lease 41 test site. These samples contained 55 to 60 weight percent, compared with 30 percent for the samples taken in 1990 by Monenco. Golder’s results indicate a material with relatively negligible toxicity.

Golder is carrying out further work with these samples to evaluate the toxicity of pore fluids released under various sequences of pore fluid replacement. Initial indications show increasing toxicity with continued pore volume displacement. The Phase I study obtained a Microtox reading of 70 percent while Phase II obtained 53 percent for the first volume replacement and about 45 percent for the second replacement. Further evaluation of these results is needed to determine the impact of the test procedure on the results and the relative significance of the deterioration.

5.6 Conclusions

- Tailings from the OSLO extraction processes can be disposed using either the conventional beaching technique (with appropriate modifications) or other emerging disposal techniques such as freeze-thaw and alternate layering of fines and sand. While this paper establishes the technical feasibility of these approaches, economic incentives need to be established for each application.
- Because OSLO fine tails settle much faster, land disturbance will be less than in current commercial operations if the conventional beaching technique is used.
- Both coarse and fine solids resulting from the two OSLO processes can be used as subsurface material for reclamation purposes.
- Further understanding of the long-term impact of the leachate generated from the containment is required.

5.7 Acknowledgments

The author would like to thank OSLO Joint Venture owners and AOSTRA for allowing this information to be included in the Fine Tails Consortium’s Information Volumes. It is recognized that both OCWE and OHWE are patented processes and that the OHWE process technology was offered for comparison test work only. It is not a part of the Fine Tails Fundamentals Consortium’s technologies patented processes.

6.0 ZERO FINE TAILS EXTRACTION PROCESS RESULTS

6.1 Introduction

In Syncrude's bitumen extraction operation, tailings are pumped either to the original Mildred Lake Settling Basin or to the newer SouthWest Sand Storage area. Coarse solids settle quickly to form a beach while fine solids remain suspended in runoff water that is subsequently returned to the Mildred Lake Settling Basin for clarification. In the settling basin, runoff water settles over time to produce a denser fine tails phase and a clarified water layer which is recycled to the extraction process. The maximum allowable solids concentration in the recycle water is 0.1 percent by weight, as determined by heat exchanger and extraction process requirements. The fine tails phase continues to densify until it forms mature fine tails which must ultimately be transferred to permanent storage below grade or otherwise reclaimed to meet environmental requirements.

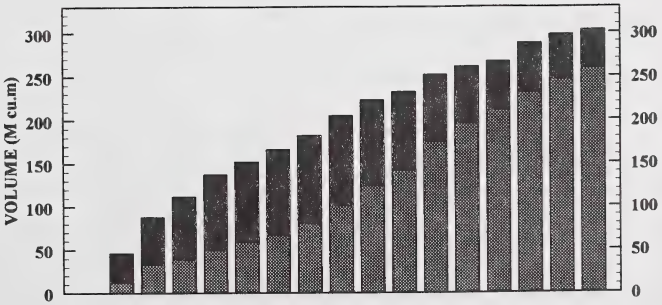
The fine tails volume accumulated in Syncrude's settling basin between 1979 to 1994 is shown in Figure 6-1 [1]. The settling basin currently contains 259 M m^3 of fine tails and 44 M m^3 of free water. Since the fine tails phase is approximately 78 percent water by volume, only 52 M m^3 of it consists of mineral particles. On an overall volumetric basis the settling basin inventory consists of 83 percent water and 17 percent mineral particles. Thus, there is considerable incentive to find ways of either reducing or eliminating the production of fine tails or of incorporating them in stable, solid deposits.

6.2 Insight From Fine Tails Formation Studies

Studies by the Fine Tails Fundamentals Consortium on the structure of fine tails and on the mechanism of fine tails formation have shown that "ultra-fine" particles (defined as less than 0.2 μm) will aggregate into flocs at a concentration of 5 grams of solids per 100 grams of water. The implication of this finding is that fine tails form as a result of larger fine particles being trapped in these flocs and prevented from settling to a denser deposit. It has been proposed by Schutte that fine tails could be avoided if individual ultra-fine particles could be either well enough dispersed to prevent flocs from forming or brought into a coagulated state [2,3].

6.3 Principle of the ZEFTE Process

In a Zero Fine Tails Extraction (ZEFTE) operation, ultra-fine particles would remain in suspension and would follow the water. The maximum size of non-settling mineral particles (including ultra-fines) that return to extraction in recycle water would depend on the dispersing agent used and on the settling areas available. As extraction tailings are discharged at the sand storage areas, sand and silt would be deposited on the beach trapping water and non-settling fines in the void spaces between sand grains. Beach runoff would be collected in a small holding basin (in lieu of a large settling basin) from which water laden with non-settling fine solids would be recycled to extraction. Some make-up water would have to be supplied to extraction to compensate for water lost to the sand deposit.



	1978		1980		1982		1984		1986		1988		1990		1992		1994
FINE TAILS VOLUME ▨		12	33	39	50	59	67	80	101	124	142	175	196	212	231	247	259
FREE WATER VOLUME ■		34	55	72	87	93	99	102	104	99	90	77	65	55	57	51	44

FIGURE 6-1. Changes in volume of clear water and fine tails zones of tailings settling basin: 1980–1994.

It is important to emphasize that water and non-settling fine minerals do not require separate storage in a ZEFTE operation. Instead they are recycled to extinction and thus are eventually all captured in the beaches. Under steady-state conditions the mass of non-settling fine solids captured in the sand deposit is equal to the mass entering the process with oil sand feed [4]. That is,

$$CT = cs$$

where C = concentration of non-settling solids in total oil sand solids, kg/kg

T = feed rate of total oil sand solids, kg/sec

c = concentration of non-settling fines in tailings water, kg/kg water

s = rate tailings water is co-deposited in the beach, kg/sec.

Since $s = yT$, where y = water in sand deposit, kg/kg oil sand minerals, then $c = (CT)/(yT) = C/y$.

That is, the steady-state concentration of non-settling fines in recycled water is directly proportional to their concentration in the oil sand and inversely proportional to the amount of water trapped in the sand dump. Note that in this simplified analysis non-settling fines leaving the process with the froth product are considered negligible and have thus been ignored.

By weight, a typical sand deposit assays 0.40/21/78.6 bitumen/water/solids, respectively. Hence, the concentration of the non-settling fines in the recycled water phase at steady-state is equal to 78.6/21 (~3.7) times their concentration in the oil sand mineral phase. This steady-state relationship can be verified experimentally.

Other issues that need to be addressed for the ZEFTE process are:

- effects of high concentrations of fine solids on extraction performance,
- long term stability of the resulting beach deposit,
- heating of the high fines process water, and
- effects of process water chemistry on downstream operations.

The work described in this paper deals only with the first issue.

6.4 First ZEFTE Pilot Program (1992)

A single 52-hour pilot run was conducted from June 2 to 4 in 1992 to demonstrate the concept of the ZEFTE process [3,5]. The test facility Syncrude Research's Experimental Extraction Circuit, and the dispersion of ultra fine particles was enhanced by using sodium silicate "N" as a process aid rather than sodium hydroxide. The chemical symbol for this is $Na_2O{\cdot}3.3(SiO_2)$.

The intent of the first program was to (1) show that fine tails generation can be prevented by keeping ultra-fines suspended in water and (2) observe the effects that a high loading of fine solids in extraction water has on process performance.

The experimental circuit used a screw classifier and a settler to recycle water from tailings to extraction. The screw classifier and the settler were used to simulate beaching and clarification of runoff water in the Mildred Lake settling basin. The classifier was used to move coarse, wet minerals away and allow water to run off carrying fine minerals with it. The purpose of the 1.8-metre settler was to accept the simulated beach runoff stream and allow the larger-sized particles to settle prior to recycling the runoff water to the extraction process.

The classifier, however, was inefficient at removing coarse particles which allowed silt and sand to pass on to the settler thereby overloading the settler underflow pump. The result was that particles as large as 22 μm were returned to the process in the recycle water. In a commercial operation recycle of sand and silt to the process would not occur. A schematic flow diagram of the experimental circuit is given in Figure 6-2.

The 125 tons of oil sand processed during the first ZEFTE program would have generated about 25 m^3 of fine tails had it been processed using the current commercial extraction process. By contrast, the water inventory at the end of this run was 7.0 m^3 (includes total process vessel volume) or 28 percent of the volume of fine tails the current commercial process would have produced. Had the run been continued longer, the water inventory would of course have been the same 7.0 m^3. Thus, the test supported our initial hypothesis that fine tails generation can be prevented.

Separation problems were, however, experienced during the first ZEFTE run and we attributed those problems to the high recirculating load of coarse solids up to 22 μm in size. A poor, indistinct interface was usually present between the froth and middlings phases in the primary separation vessel, and the bitumen content of froth product deteriorated during the course of the run from 65 to 45 percent by weight. Total bitumen recovery, however, remained constant at about 90 percent throughout the test.

6.5 Second ZEFTE Pilot Program (1993)

The purpose of the second pilot program [6–10] was to determine if poor froth quality could be avoided in a ZEFTE

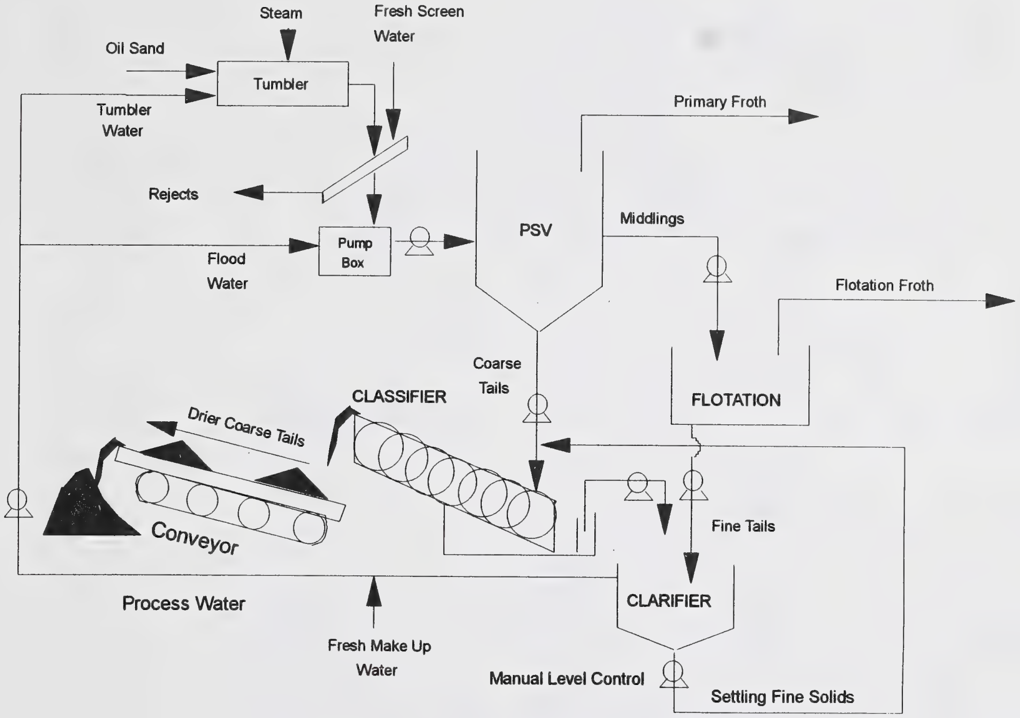


FIGURE 6-2. Process flow diagram for first ZEFTE pilot test.

process. As in the first program, the test circuit simulated extraction and beaching operations. However, we added a scroll centrifuge to remove particles larger than 2 μm from beach runoff and thus to prevent silt from being recycled back to the process.

6.5.1 Experimental Details

This test was also conducted on Syncrude Research's Experimental Extraction Circuit which consisted of a tumbler, a primary separation vessel (PSV) and a flotation machine.

Baseline runs were carried out to provide a benchmark against which results of the ZEFTE experiments with sodium silicate could be compared. The baseline runs consisted of running the circuit in the current commercial process configuration at various NaOH addition rates to determine the dosage that maximized the recovery of bitumen from the oil sand we were processing.

With the ZEFTE process configuration flotation froth was recycled to the PSV to provide a single froth product. ZEFTE tailings were sent to a flume measuring 7.9 m long × 2.2 m wide × ~1.5 m deep for deposition of coarse solids. A small runoff pond was maintained at the discharge end of the flume to help reduce the amount of coarse solids returning with the runoff water. The flow rate of beach runoff and the size of the recycle pond were controlled by a weir arrangement constructed with sand bags. As the beach deposit grew, the sand bag dam was slowly raised to maintain a steady flow of runoff from the beach deposit. Water and fines overflowing the dam were collected in a mixing drum and pumped to a scroll centrifuge for additional removal of solids prior to recycling water and non-settling solids to the extraction process. The scroll centrifuge was also used, as necessary, to adjust the dryness of the centrifuge underflow. A schematic flow diagram for the ZEFTE experiment is given in Figure 6-3.

Water holdup in the extraction and tailings circuit was:

• PSV	0.534 m^3
• Flotation	0.191 m^3
• Runoff pond	0.360 m^3
• Centrifuge feed sump	0.110 m^3
• Total	1.195 m^3

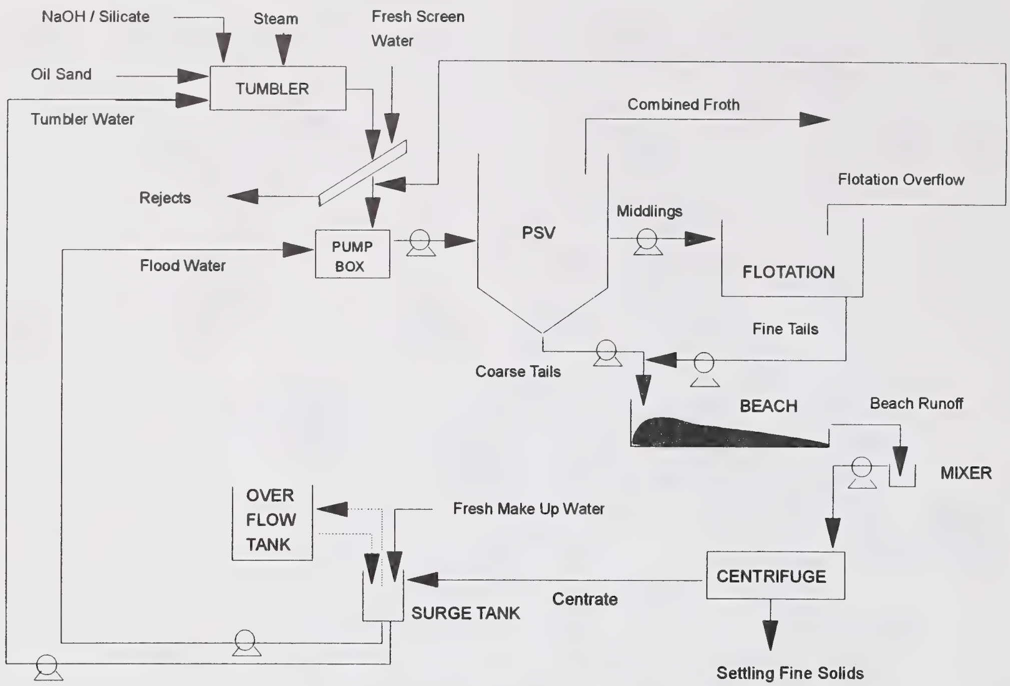


FIGURE 6-3. Process flow diagram for second ZEFTE pilot test.

With an oil sand feed rate of three tph and with about ten hours of running time, six system residence times were available to allow fine solids to reach a steady-state concentration in the recycle water stream.

All stream samples were analyzed for oil, water, and solids content (OWS). The X-ray Disc Centrifuge method was used to determine the particle size distribution (PSD) of the solids.

6.5.2 Measurements and Sampling

Beach Deposit Profile — The compositions of extraction tailings, beach deposit, and beach runoff streams were determined by sampling them. At the end of the test the beach was allowed to drain for one hour then samples were taken from three locations along the beach at distances of 1.5 m, 3.0 m, and 4.5 m from the feed end. These samples were taken just below the surface of the deposit with as little disturbance to the deposit as possible so that the fines and water contents would be representative. Finally, both the slope angle and volume of the beach deposit were estimated.

Settling Columns — Twenty litre samples of the combined primary and secondary tailings stream were collected for each of the three test conditions. Combined tailings were used instead of runoff because the method of controlling weir height by adding or removing sand bags could result in large variations in runoff flow rate and composition. The tailings samples were remixed and poured into Plexiglas columns 1.2 m high by 150 mm in diameter for subsequent observation and measurements. Samples for OWS and PSD analyses were collected from the tailings while the columns were being filled and later from the fine tails phase in the columns after 166 days of settling. Throughout the observation period we recorded the location of the interfaces between clarified water and fine tails and between fine tails and sand.

6.5.3 Test Conditions

A number of test conditions were evaluated, but only the baseline run and the three ZEFTE runs are reported. The baseline run on June 24 established an optimal NaOH dosage of 0.05 weight percent on oil sand. The ZEFTE test on June 29 used 0.03 weight percent NaOH on oil sand, with the remaining 0.02 weight percent being replaced with the mole equivalent of sodium silicate. The dosages of the process aids for all four runs are summarized in Table 6-1 below.

TABLE 6-1. Process aid dosages.

Date	Process	Process aid (wt% on oil sand)	
		NaOH	Na silicate 'N'
June 24	Conventional	0.05	–
June 29	ZEFTE	0.03	Mol equivalent of 0.02 NaOH
July 7	ZEFTE	–	Mol equivalent of 0.02 NaOH
July 8	ZEFTE	0.03	–

Notice that both the July 7 and July 8 tests used less than the optimal chemical dosage in terms of mole equivalent of caustic. However, both tests were operated with a PSV froth interface underwash feature which consumed a portion of clean make-up city water.

6.5.4 Results and Discussion

Beach Deposit Slope — In beaching tailings into the flume we succeeded in forming a beach of coarse sand particles which also captured a portion of the fine solids present in the tailings. Beach slopes of 7 percent were obtained in the flume for all ZEFTE tests. Because of higher fines in ZEFTE process streams including the tails deposit, we were expecting shallower slopes such as were observed in a 1990 field test in which extraction tails were spiked with fine tails [11] in order to increase fines capture. In any event shallower slopes would be expected in a commercial operation, and we attribute these rather steep experimental slopes to low flow velocities and flume wall effects.

An internal comparison of ZEFTE tests shows that, in comparison to caustic, using sodium silicate as a process aid has very little influence on the slope of tails deposits. The geotechnical stability of ZEFTE slopes, however, is an issue that needs to be addressed in a future study.

Fines content and percentage of fines capture in our experimental ZEFTE beach deposits were higher than for deposits formed by the current commercial extraction process. The fines in the inflow to the beach remained fairly consistent throughout the test period indicating that a steady-state fines concentration had been achieved in the system.

Settling Column Test — The intent of the ZEFTE process is that sub-micron particles in tails will be recycled and continually captured in the sand deposit Because of this batch settling tests that permit the formation of a coarse solids phase with a fine tails phase above cannot simulate the ZEFTE process. Nevertheless these tests were included in the second ZEFTE program to illustrate the effects that sodium silicate and sodium hydroxide have on fine solids (when they are present in high concentrations typical of the ZEFTE process) and to bolster our hypothesis that the formation of fine tails results from failure to adequately disperse ultra fine solids in the first place.

Solids Content and Particle Size — The results from the settling tests conducted in the Plexiglas columns showed that after 166 days, the solids content in the fine tails phase were twice as high in the NaOH test as compared to the silicate tests (Figure 6-4). The grains size distribution (Figure 6-5) shows that 60 percent of the solids in the fine tails phase for the NaOH phase were larger that 0.2 μm as compared to only 10 percent in the silicate tests. These results support the hypothesis that the use of silicate in place of NaOH as the process extraction aid will significantly reduce the amount of fine tails production.

Interface Monitoring — Observations of the interfaces between the clear water layer and the fine tails phase and between the fine tails phase and the coarse sand phase showed that when NaOH was used as a process aid, the fine tails phase settled faster. After 166 days, the change in the height of the water/fine tails interface was –2.5 percent for NaOH *versus* –1.25 percent for silicate. The NaOH produced a distinct interface between the fine tails and coarse sand phases. The sodium silicate produced an undefined, fuzzy interface between the fine tails and coarse sand phases that initially increased in height as the sand slowly settled and then later decreased as the sand bed consolidated. After 166 days, the change in the height of the fine tails/sand interface was –55 percent for NaOH *versus* –51 percent for silicate. These tests show that when sodium silicate is used as a process aid, it is effective in keeping fines in suspension while allowing more coarse solids to settle (albeit at a slower rate than when NaOH is used).

Extraction Performance — The oil sand feed composition was constant throughout the experimental runs averaging 10.7 percent bitumen, 5.3 percent water, and 84.0 percent solids by weight. Mineral particles less than 44 μm in size were 23 weight percent of the total solids.

As previously demonstrated the weight percent of non-settling fines in recycle water under steady-state operating conditions should be directly proportional to the weight percent of non-settling fines in oil sand minerals and inversely proportional to the water and solids ratio in the sand deposit.

For the second ZEFTE pilot program solids in the recycled water were all less than 1 μm in size. Solids smaller than this in the oil sand feed averaged 3.53 weight percent of total oil sand solids, and the water-to-solids ratio in beach samples for the three ZEFTE experiments ranged from 0.28 to

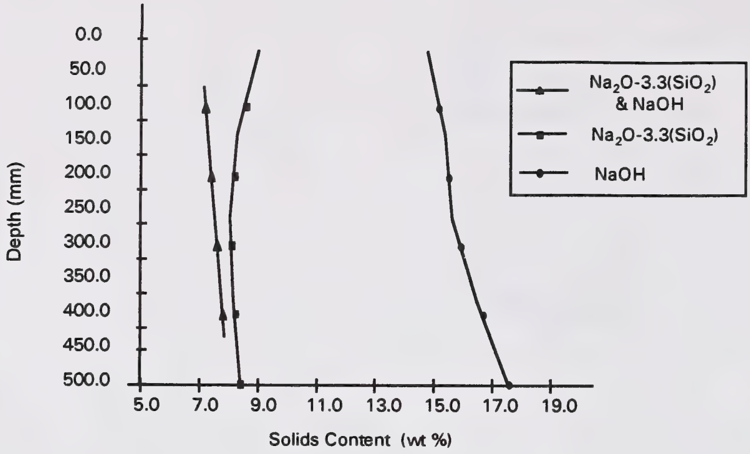


FIGURE 6-4. Solids content in ZEFTE fine tails after 166 days.

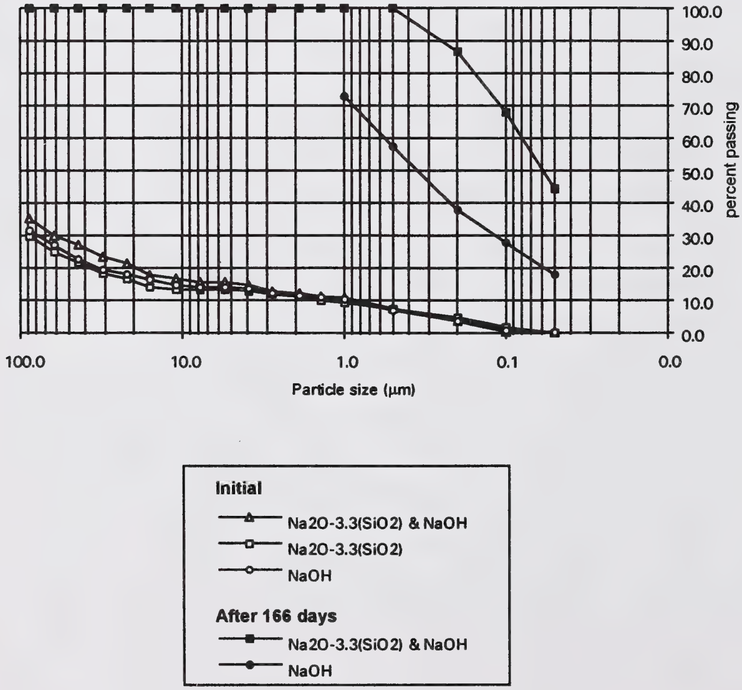


FIGURE 6-5. ZEFTE settling columns — particle size curve.

0.29 wt/wt. By material balance, then, the concentration of non-settling minerals in the recycled water should have been 12.1 to 12.6 percent by weight. Measured concentrations were 10.0 percent for the test which used a combination of sodium hydroxide and sodium silicate, 11.74 percent for the test using only sodium silicate, and 12.88 percent for the test using only sodium hydroxide.

As Figure 6-6 shows bitumen recoveries for all three ZEFTE experiments were higher than for the baseline case. The best bitumen recovery (on a reject-free basis) for the ZEFTE process was 98 percent for the test which used NaOH as the process aid and utilized a froth/middlings underwash. The baseline run (using NaOH) had the lowest bitumen recovery — just 89 percent on the same basis.

Because beach runoff is recycled, it is plausible to assume that bitumen recovery for the ZEFTE process should be higher because losses would be limited to bitumen contained in water trapped in the voids between sand grains in the beach. In practice of course, it is possible for bitumen mats to form in the small runoff catch basin and either sink or cause difficulties with the recycle pumps.

As Figure 6-7 shows, the combined froths for all three ZEFTE tests were higher in bitumen content than froth from the baseline run. Combined froth for the ZEFTE test using NaOH and the froth/middlings underwash was around 69 weight percent bitumen, whereas combined froth for the baseline run was around 63 percent bitumen. Also, the lowest froth solids assays were obtained for the ZEFTE process operating with froth/middlings underwash.

The solids content of the middlings stream for the baseline case, in which beach runoff was not recycled and the make-up water was 100 percent clean city, was typically around 27 percent by weight. For the ZEFTE tests in which beach runoff was reused directly, and in which make-up water consisted of 30 percent city water and 70 percent pond water from Syncrude's commercial operation, the solids content of the PSV middlings stream was high at 36 percent by weight. Yet in spite of this, bitumen recoveries did not suffer. This is attributed to the use of an appropriate caustic-silicate combination in the one case and to the introduction of clean make-up water under the PSV froth/middlings interface in the other two cases.

Flotation tailings in the ZEFTE tests contained more than 40 weight percent solids. For the baseline case flotation tailings contained less than 30 percent solids.

Since relatively few oil sand solids report to the froth product the solids deposited on the beach are essentially the total solids entering and leaving the ZEFTE process. ZEFTE beach deposits assayed 0.2–0.7 percent bitumen and around 75 percent solids by weight.

As previously described beach runoff water (~2 percent bitumen, 16 percent solids by weight) was fed through a scroll centrifuge for more complete removal of settling solids. Underflow from the centrifuge contained 60–65 solids by weight and its mass flow rate was calculated by material balance to be around 12 percent of the beach runoff flow rate. Centrate containing 11 weight percent solids and 2 weight percent bitumen was added to make-up city water and used as

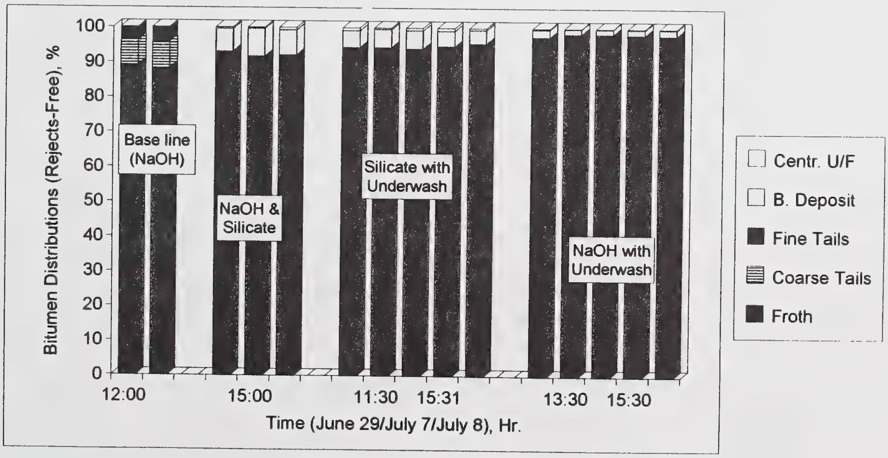


FIGURE 6-6. Bitumen distribution (MATBAL).

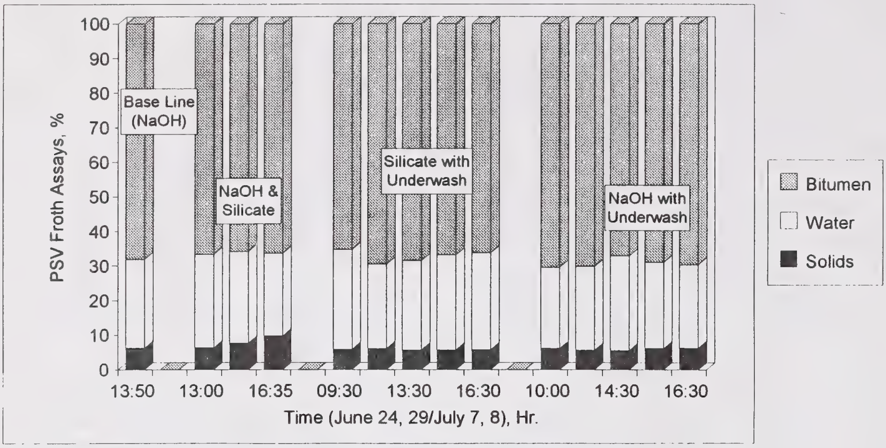


FIGURE 6-7. PSV froth composition (MATBAL).

tumbler water and flood water for extraction. The make-up water used was equal to the water trapped in the beach deposit. Tumbler and flood waters for the ZEFTE runs contained about 8 percent solids and 1.5 percent bitumen by weight.

6.6 Summary

Combined tailings from Experimental Extraction Circuit runs were successfully beached into a flume and the runoff collected and recycled to the process. Beaching tailings into the flume was effective in capturing most of the sand and a portion of the fines in the resulting deposit.

Experimental beach slopes obtained in the flume used for the second ZEFTE program were higher than slopes measured during a 1990 field test in which extraction tails were spiked with fine tails in an attempt to increase fine tails capture. This discrepancy is likely attributable to smaller flow velocities in the pilot tests and wall effects from the flume. Restricting our comparison of slope angles to the results of the ZEFTE pilot runs, the choice of process aid appears to have little or no effect on the slope of tailings deposits. The geotechnical stability of the deposits is an issue that was not directly addressed in this study and needs to be addressed more fully in a future study.

Experimentally, fines content and fines capture in beaches formed by the ZEFTE process were higher than in the beach formed by the current commercial process. The fines in the beach inflow remained fairly constant throughout the test period. Furthermore, the concentration of the non-settling fines in the recycled water phase reached their steady-state value at ~3.7 times their concentration in the oil sand mineral phase. Hence, the steady-state fines concentration was achieved in the system for the ZEFTE experiments.

The test program described in this paper demonstrated that the extraction process can be operated using reclaim water with a much higher fine solids content (>8 percent) than prescribed by water quality criteria (~0.1 percent) for Syncrude's commercial operation. Thus it would appear that current water quality criteria are set entirely by considerations related to fouling of heat exchangers not process performance.

The three ZEFTE processes, all tested on the same oil sand, gave higher bitumen recoveries than the baseline extraction process with reduced chemical consumption and no froth quality penalty despite higher steady-stated levels of fine solids in process streams.

These results were achieved by using clean make-up water as a the froth/middling underwash in the PSV.

Alternately, the ZEFTE process can produce extraction performance similar to the baseline process by omitting the froth/middlings underwash and using equivalent dosages of an appropriate combination of caustic and sodium silicate.

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7.0 THE BITMIN PROCESS

7.1 Background

Bitmin Resources Inc. was formed by Fording Coal and Kilborn Inc. in 1992 to develop the Countercurrent Desander (CCDS) process. The CCDS process uses warm water to digest and condition oil sand and uses relatively less thermal and electric energy than other known aqueous processes. Unlike the Clark Hot Water Extraction process, the CCDS process uses no sodium hydroxide so tailings and tailings waters are relatively non-toxic. Another major advantage is that tailings are trafficable and can be returned to the mined-out pit as the production proceeds.

Process concepts about oil sand disaggregation, tailings solids rejection, and bitumen recovery were confirmed first in bench-scale experiments and later in pilot plant test work at the Alberta Research Council's (ARC) pilot facility in Calgary, Alberta. Continuous pilot runs on three different oil sands ranging in grade from 8 to 13 percent bitumen resulted in over 90 coherent mass balances that confirmed acceptable recovery, characterized recovered bitumen and rejected solids, and produced design information for a larger scale demonstration plant.

In 1993 a 20 tonnes-per-hour demonstration plant was constructed on the Suncor oil sand lease 35 kilometers north of Fort McMurray, Alberta. From August to December of 1993 operating techniques were developed, mechanical design factors were confirmed, and laboratory testing of filtration and thickening steps was carried out. Between December 1993 and February 1994 the demonstration plant was modified to allow co-filtration of all plant tailings. In all, more than 200 coherent mass balances were obtained from processing 10,000 tonnes of oil sand. Eight different lots of oil sand were processed ranging in bitumen content from 7 to 13 percent by weight and in fines content from 35 to 4 percent.

The latest stage of development of the CCDS process occurred in spring of 1994 when about 15,000 tonnes of oil sand was processed to obtain mechanical design data for commercial application of the process. Another purpose of this stage was to obtain data that would permit forecasts of bitumen recovery, froth quality, and geotechnical properties of tailings for specific ore bodies.

7.2 Process Description

Figure 7-1 is a simplified flow sheet of the 20-tonnes per hour demonstration plant just described. Mined oil sand passes through a Stammler feeder-breaker in order to limit maximum lump size in the feed to 20 centimeters. From the feeder-breaker oil sand is conveyed to the Strand Separator where bitumen is separated from mineral solids by gentle agitation in warm water flowing countercurrent to the sand. For testing purposes, process temperatures were varied from 43°C to 58°C during the demonstration runs and water-to-oil sand ratios were varied from 0.7:1 to 1.5:1. Generally, low temperatures and low water-to-oil sand ratios gave the best results.

From the Strand Separator desanded slurry flows to a circuit consisting of a froth separator, an induced air flotation machine, and a froth cleaner for recovery of bitumen from water and fine solids. Typically, bitumen recovery of about 91.5 percent can be expected from an oil sand with 11 percent bitumen in it. Cleaned froth will assay 55 – 60 percent bitumen and 7 – 10 percent solids by weight.

Tailings from the flotation machine flow to a conventional mineral thickener where fine solids are thickened with the assistance of an anionic polymer flocculant. Flocculant is consumed at the rate of about 35 grams per tonne of oil sand. From the thickener clarified water is mixed with fresh make-up water and heated in a submerged flame water heater with a heat requirement of about 80 Joules per gram of oil sand (compared with about 250 Joules for hot water processes).

Underflow from the thickener is mixed with flocculant and damp solids from the Strand Separator prior to being filtered on a horizontal belt filter. Filtration of this mixture of coarse sand and thickener underflow is referred to in this paper as "co-filtration." Typical moisture contents of the filter cake range from 8 – 15 percent by weight. Higher moisture contents are typical of oil sands with a high fraction of fine solids in their mineral matrix.

During the demonstration runs Cytec Superfloc 1206™ was used to assist thickening and filtration. While it is not shown on the flow sheet, sulfuric acid is used in the CCDS process to neutralize bicarbonate from recycle water. The sources of bicarbonate ion are the oil sand feed and combustion products from water heating. When it builds up to more than 350 ppm, flocculation becomes difficult and process water will not clarify in the thickener.

7.3 Tailings Production

As described earlier a significant advantage of the CCDS process is that the tailings can be transported by truck and are trafficable. The following aspects of the process make this advantage possible.

7.3.1 Minimal Size Reduction

Clay lenses are kept as large as possible by setting the feeder-breaker so that oil sand is not broken any more than necessary to transport it on conveyors.

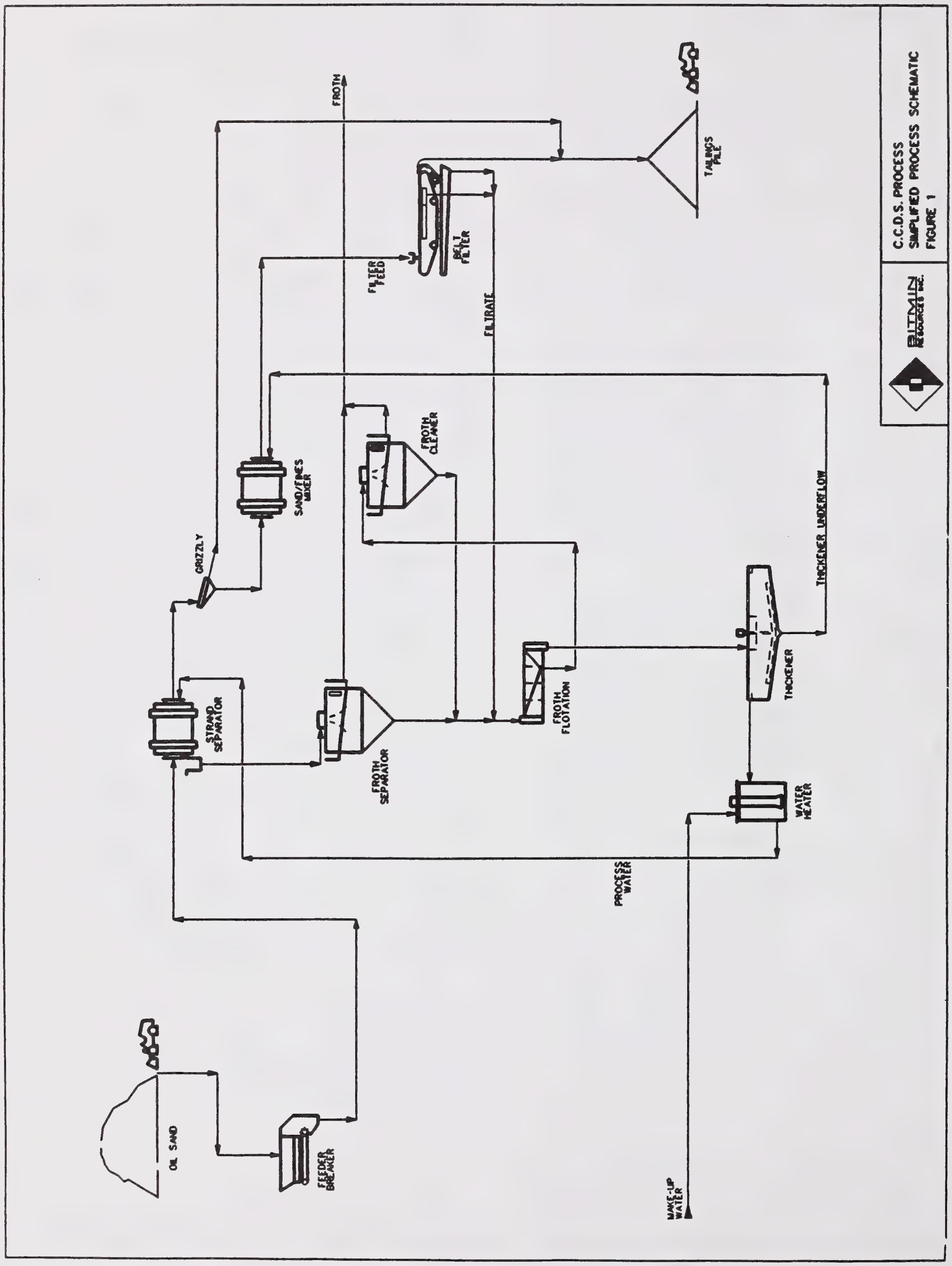


FIGURE 7-1. Flow sheet of Bitmin process.

7.3.2 Gentle Digestion

Disaggregation of oil sand and separation of coarse solids from bitumen is done in the Strand Separator. Warm water flowing countercurrent to the sand softens bitumen allowing sand to be liberated from the bitumen matrix. Coarse solids and clay lumps are mechanically propelled along the separator and expelled as a partially drained solid. Separated bitumen, water, and some fine solids form a slurry that exits the front of the Strand Separator. Mechanical agitation is closely controlled in this device by variable height lifting agitators that minimize mechanical breakage of clay lenses.

7.3.3 Thickening of Fine Tailings

After the removal of bitumen the slurry is thickened in a typical mineral thickener. Because clay minerals are not dispersed with caustic soda, quite high underflow densities are possible. Solids contents of 35 – 45 percent by weight are typical. It is important not to use too much sulfuric acid during water treatment because pH levels below 6.0 limit underflow densities to about 25 percent solids. Thickeners of a size required for commercial production are readily available.

7.3.4 Filtration of Total Tailings

All coarse and fine solids leaving the CCDS process are combined and filtered on a horizontal belt vacuum filter to reduce moisture content to 15 weight percent or less. This step is what makes CCDS tailings truckable and trafficable. Filters of a size required for commercial production are readily available.

7.4 Disposal of Total Tailings

All tailings produced during the 20 tonnes per hour demonstration runs were transported with a five cubic yard John Deere 744E front end loader to a large ramp constructed entirely of tailings. This ramp was from all of the tailings produced from the processing of 15,000 tonnes of oil sand. A section through this ramp is shown in Figure 7-2.

The co-filtered tailings were deposited in a ramp which rises from the top of a berm, at an angle of 10 degrees, reaching an ultimate height of nine metres. The ramp was constructed by driving the loader up the ramp and dumping on the advancing face.

During placement, the co-filtered tailings behaved like a normal moist silty fine sand. As the tailings were dumped on the advancing ramp face they would initially over steepen the upper portion of the slope and then, with on-going placement, a local failure would occur flattening the slope to a stable configuration. The slope of the advancing face varied somewhat during construction of the ramp with measured slope angles ranging from 32 to 40 degrees.

A small portion of tailings, produced when oil sand fines contents exceeded 20 percent of total solids, was too moist for immediate placement. This material was set aside for 24 to 48 hours and then placed on the ramp.

7.5 Geotechnical Properties of Filtered Tailings

Several standard laboratory tests were performed on tailing products from the demonstration plant in order to define their geotechnical characteristics. In the discussion which follows the term "fines" always means solids passing a 325 mesh screen, that is, solids less than 44 μm in size.

7.5.1 Shear

Direct shear tests were performed on co-filtered tailings samples obtained from processing three oil sand lots. Peak phi angles ranged from 32 to 36 degrees, while residual phi angles ranged from 28 to 31 degrees. These strength values are consistent with those expected of a fine sand containing some fines. After removing clay lumps from these samples their fines contents ranged from 7 to 12 weight percent of total solids. Unexpectedly, the samples with higher fines (after clay lump removal) had greater strength. The flocculants used in the process did not appear to significantly effect the shear strength of the tailings.

7.5.2 Segregation

Bulk samples of filtered sands, thickened fine tails, and process water were used for segregation tests. These samples were combined, agitated and then observed to determine if segregation would occur as they settled. Settlement of mixtures occurred rapidly and was complete in 48 hours. Settlement of thickener underflow was essentially complete in 96 hours. The expelled water layer that formed on top of the solids was essentially clear in 24 to 96 hours. The purpose of this testing was to gain insight into the behavior of Bitmin tailings if disposed of as a slurry.

None of the mixtures of filtered sand and thickener underflow exhibited any significant segregation. At the beginning of settling these samples ranged from 42 to 62 weight percent total solids. Fine solids ranged from 28.3 to 16 weight percent of total solids. After settling was complete, the solids content of the deposit beneath the clear water layer ranged from 65 weight percent for settled thickener underflow to 75 weight percent for a mixture consisting of one part sand to two parts thickener underflow, by volume.

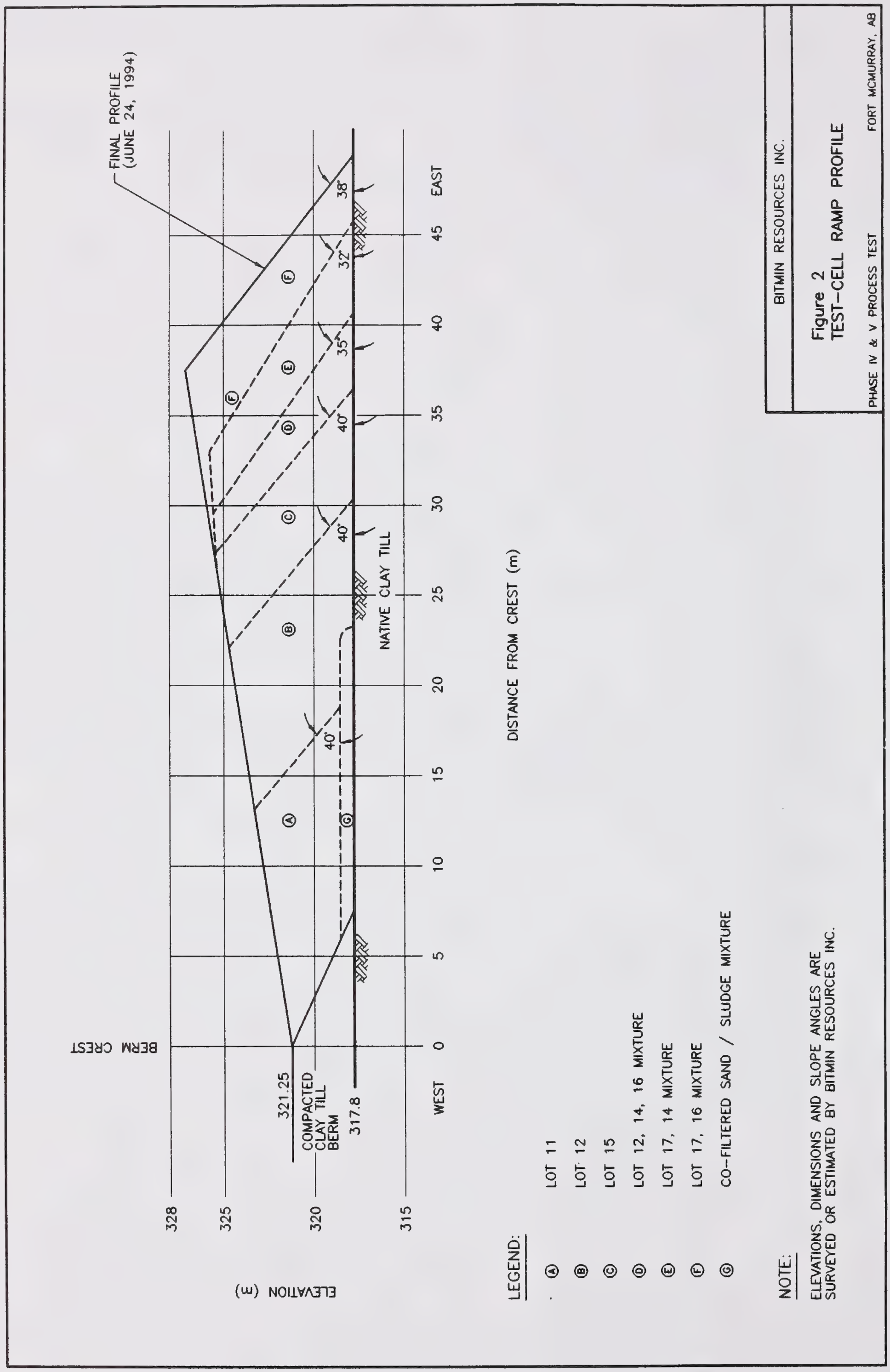


FIGURE 7-2. Tailings ramp.

Samples thickener underflow and thickener underflow with a small amount of added sand that were diluted with process water to an initial solids concentration of 30 percent by weight did segregate into coarse and fine tailings phases.

7.5.3 Permeability

Permeability tests were performed on both filtered sand and co-filtered tailings. Twelve of the tests were standard laboratory percolation tests, while eight other tests were done in large test tanks. The permeability of co-filtered tailings ranged from 4×10^{-4} to 4×10^{-4} cm/sec. This range of permeability is significant in that the upper end is typical of relatively free-draining material and the lower end of relatively poor-draining material. Markedly different drainage characteristics can be expected of deposits constructed of material at one or the other end of these extremes.

Both permeability and in-plant filter tests indicate that cake with 15 weight percent moisture cannot be produced if fines in the tailings stream exceed 18 percent of total solids. A portion of thickener underflow will have to be disposed of separately when processing oil sand with more than 18 percent fines. This will not be a Serious problem as thickener underflow is non-toxic and this stream will constitute only a small portion of total tailings for most commercial ore bodies. Permeability results are given in Table 7-1.

7.5.4 Load Bearing

Standard Proctor tests (ASTM D698) were carried out on samples of co-filtered material from different ore lots. These tests determined a wide range of maximum dry densities ranging from 1650 kg/m^3 to greater than 1900 kg/m^3 and optimum moisture contents ranging from 10 to 14 percent by weight.

California Bearing Ratio (CBR) tests were done on three samples to assess trafficability. The results indicate that co-filtered tailings near optimum moisture will be suitable as

TABLE 7-1. Permeability tests results.

Sample	Lot #	Test type	Tailings material	Average permeability (cm/sec)	Percent* fines
Perc-1	2	Percolation	Filtered sand	1.6×10^{-5}	10.1
Perc-2	2	Percolation	Filtered sand	8.3×10^{-6}	31.2
Perc-3	11	Percolation	Filtered sand	7.1×10^{-6}	17.6
Perc-4	12	Percolation	Filtered sand	1.6×10^{-6}	11.5
Perc-5	12	Percolation	Co-filtered	2.6×10^{-4}	7.9
Perc-6	15	Percolation	Co-filtered	3.0×10^{-4}	4.5
Perc-7	15	Percolation	Filtered sand	2.6×10^{-4}	5.0
Perc-8	13	Percolation	Co-filtered	2.3×10^{-5}	19.2
Perc-9	12/16	Percolation	Co-filtered	4.7×10^{-4}	14.1
Perc-10	16	Percolation	Co-filtered	$1.O \times 10^{-5}$	16.2
Perc-11	14	Percolation	Co-filtered	2.7×10^{-4}	8.4
Seg-3	8	Percolation	Filtered sand	1.8×10^{-6}	28.6
Perm-1	2	Tank permeability	Filtered sand	2.0×10^{-5}	6.3
Perm-2	2	Tank permeability	Filtered sand	2.3×10^{-6}	9.1
Perm-3	12	Tank permeability	Co-filtered	1.6×10^{-4}	7 5
Perm-4	15	Tank permeability	Co-filtered	3.6×10^{-4}	3.8
Perm-5	13	Tank permeability	Co-filtered	1.5×10^{-5}	14.7
Perm-6	12/16	Tank permeability	Co-filtered	5.3×10^{-5}	11.4
Perm-7	16	Tank permeability	Co-filtered	6.1×10^{-6}	19.3
Perm-8	14	Tank permeability	Co-filtered	1.0×10^{-4}	9.3

* Passing #325 sieve, clay lumps removed.

subgrade for mine haul roads, providing that the material is contained and suitably capped with other materials such as overburden.

7.5.5 Penetration

Eleven Cone Penetration Tests (CPT) were done on the tailing ramp, and then eleven test holes were drilled and 34 Standard Penetration Tests (SPT) were performed at various depths in the holes.

The results of the CPT program indicate normalized peak tip resistance values between 3.5 and 14 MPa at depths between 0.5 and 1.0 metres below ramp surface. Below 1.0 meter the tip resistance drops rapidly to an average value of 2.0 MPa until the underlying native clay till is penetrated. Occasional spikes occur, likely due to clay lumps in the tailings matrix. These results indicate that the tailing material placed was loose except for the zone within about 1 metre of the ramp surface that had been compacted by loader activity.

7.6 Environmental Characteristics of Tailings and Drainage Water

7.6.1 Soil Toxicity Testing

Standard soil seed germination tests and earthworm survival and reproduction tests were performed on five samples of co-filtered tailings and the results are given in Table 7-2. By definition the LC_{50} level is the concentration of a test treatment that is fatal to 50 percent of the test organisms. An LC_{50} 100 means the test material is relatively non-toxic. Also by definition, the NOEC level is the lowest concentration level of a test treatment that has no effect on a test population.

7.6.2 Water Toxicity

The following four standard water toxicity tests were performed on seven sets of samples of drainage water from the tailings ramp: rainbow trout 96-hour survival, luminescent bacteria (Microtox), Ceriodaphnia survival, and Ceriodaphnia reproduction. The samples were collected from a gathering system which excluded all other sources of water. Results of the drainage water toxicity tests are given in Table 7-3. By definition the IC_{50} level is the concentration level of a test treatment that incurs a 50 percent reduction in the rate of reproduction of Ceriodaphnia.

7.7 Commercial Vision

- Because the CCDS process uses very little thermal or electric energy it is an attractive option for either remote bitumen producers or integrated producers of synthetic crude oil.
- The CCDS plant will be located closer to the oil sand mine to minimize haulage distances for both oil sand and the tailings being returned to the mined-out pit.
- Over 90 percent of the tailings produced will be returned to the mined-out pit by large (240 ton) haulage trucks and dumped on benches up to 30 metres thick. Tailings and overburden can be dumped together if it is economical to do so.
- When the plant is processing oil sand with more than 18 weight percent fines some of the underflow from the thickener will be pumped to containment pits left for that purpose. This will amount to less than 10 percent of the tailings produced. The storage areas thus created will become lakes and will improve the bio-diversity of the

TABLE 7-2. Tailings sand toxicities.

Ore type	Date collected	Seedling emergence test (acute)	Earthworm survival test (acute)
Ore # 12	04/18/94	EC_{50} >100, NOEC = 50 (not toxic)	LC_{50} >100, NOEC = 50 (not toxic)
Ore # 15	06/01/94	EC_{50} = 82 NOEC = 50 (mild reduction)	LC_{50} >100, NOEC = 100 (not toxic)
Ore # 16	06/01/94	EC_{50} >100, NOEC = 50 (not toxic)	LC_{50} >100, NOEC = 100 (not toxic)
Ore # 17	06/23/94	EC_{50} >100, NOEC = 12.5 (not toxic)	LC_{50} = 87, NOEC = 50 (relatively non-toxic)
Ore # 17	06/23/94	EC_{50} >100, NOEC = 50 (not toxic)	LC_{50} = 72, NOEC = 50 (relatively non-toxic)

TABLE 7-3. Drainage water toxicities.

Date	Rainbow trout 96-hr survival (acute)	Luminescent bacteria (acute)	Ceriodaphnia survival (acute)	Ceriodaphnia reproduction (chronic)
06/25/94	LC_{50} >100 NOEC = 100 (not toxic)	94 percent of control level (not toxic)	LC_{50} >100 NOEC = 100 (not toxic)	IC_{50} >100 NOEC = 50 (mild effect)
06/01/94	LC_{50} >100 NOEC = 100 (not toxic)	115 percent of control level (not toxic)	LC_{50} >100 NOEC = 100 (not toxic)	IC_{50} >100 NOEC = 100 (not toxic)
06/17/94	LC_{50} >100 NOEC = 100 (not toxic)	112 percent of control level (not toxic)	LC_{50} >100 NOEC = 100 (not toxic)	IC_{50} >100 NOEC = 100 (not toxic)
06/23/94	LC_{50} >100 NOEC = 100 (not toxic)	111 percent of control level (not toxic)	LC_{50} >100 NOEC = 100 (not toxic)	IC_{50} >100 NOEC = 50 (mild effect)
06/29/24	LC_{50} >100 NOEC = 100 (not toxic)	85 percent of control level (not toxic)	LC_{50} >100 NOEC = 50 (not toxic)	IC_{50} >100 NOEC = 50 (mild effect)
07/25/94	LC_{50} >100 NOEC = 100 (not toxic)	96 percent of control level (not toxic)	LC_{50} >100 NOEC = 50 (not toxic)	IC_{50} >84 NOEC = 50 (mild effect)
09/05/94	LC_{50} >100 NOEC = 100 (not toxic)	92 percent of control level (not toxic)	LC_{50} >100 NOEC = 50 (not toxic)	IC_{50} >100 NOEC = 25 (mild effect)

reclaimed landscape. This is possible because the CCDS tailings materials are non-segregating and non-toxic.

- Both in-pit and out-of-pit tailing disposal areas will be reclaimed as soon as final elevations are reached. The reclaimed areas will be returned to community use as soon as they are clear of active mining. There is minimal long term environmental liability associated with these deposits because they are non-toxic and geotechnically stable.

7.8 Acknowledgment

The author wishes to acknowledge Thurber Engineering Ltd., Edmonton, for their assistance in determining the geotechnical characteristics of CCDS tailings.

PHOTO 7-1. Co-filtered tailings being discharged from the demonstration plant.

PHOTO 7-2. Co-filtered tailings being dumped on the ramp.

PHOTO 7-3. John Deere loader on the ramp.

8.0 PROPERTIES OF ATP TAILINGS

8.1 Introduction

UMATAC Industrial Processes and Alberta Oil Sands Technology and Research Authority (AOSTRA), now a part of the Oil Sands and Research Division of the Alberta Department of Energy (AEOSRD), have developed the AOSTRA Taciuk Process (ATP) for recovering hydrocarbon from oil sand. From the start, the incentives for developing ATP technology have been both economic and environmental. In the latter case, because the process produces dry tailings, a dry landscape reclamation should be possible.

Since the ATP's development in the mid-seventies, and until recently, economic feasibility studies were based on dry tailings disposal schemes involving either trucks or conveyors and stackers. Hydraulic disposal, although more economical, was not considered due to the perception that introducing a tailings pond would negate a major environmental advantage of the process.

In 1992 a 5-tonne/hour continuous demonstration plant was commissioned on a site in southeast Calgary, and an Industry Task Force was set up to evaluate the technical and economic merits of the ATP. As a part of the optimization of ATP economics, it was decided to include hydrotransport as one of the options the Task Force would study. In defining the scope of work, it was noted that high temperatures in the ATP processor could irreversibly alter kaolinite clays and that this should improve the geotechnical properties of hydraulically transported ATP tailings, at least in comparison with fine tailings from the conventional hot water process. Consequently, a test program was structured to substantiate this observation, and Golder Associates Ltd. was commissioned to obtain geotechnical data on dry and hydraulically disposed ATP tailings for a commercial feasibility study.

The results from the tailings program and the subsequent feasibility study confirmed the geotechnical and economic advantages of hydraulically transporting ATP tailings. The program also confirmed that hydraulically disposed tailings would not require long term storage/settling ponds and that landscape reclamation similar to dry disposal (trucking) could be feasible.

This paper summarizes the results from the ATP Tailings Program which established a new direction for the management of ATP tailings solids.

8.2 Process Description

The ATP process is a continuous, flow-through process that thermally separates bitumen from oil sands and partially upgrades it, as depicted in Figure 8-1. The rotary kiln-like processor heats oil sand through various stages, reaching a maximum temperature of about 750°C. Hydrocarbon vapors are removed and subsequently recovered. At the end of the process, hydrocarbon-free tailings are quenched by spraying them with water to which flue gas bag house fines have been added.

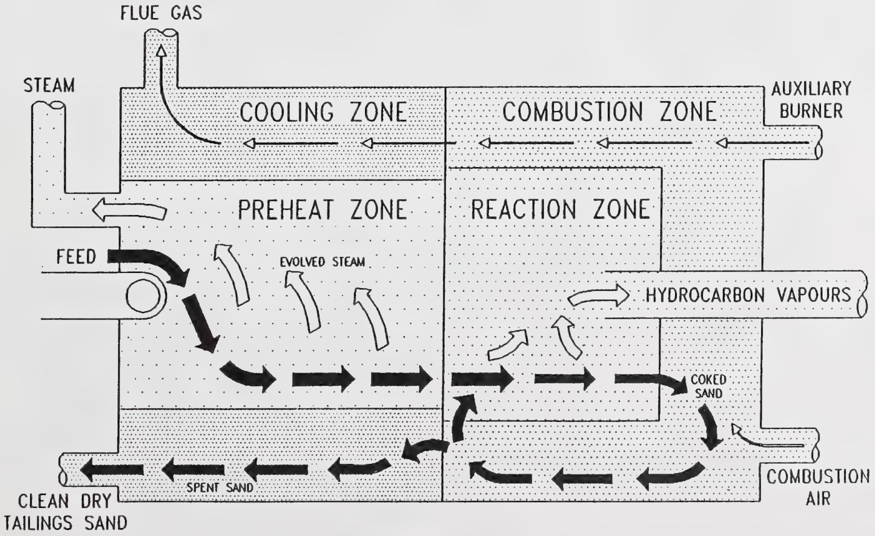


FIGURE 8-1. Simplified ATP processor schematic.

ATP tailings can be disposed of by either dry or wet methods. Dry disposal could be based on truck or conveyor transport of untreated tailings to the disposal site. Wet disposal could involve mixing the tailings with water and transporting them to the disposal site *via* a slurry pipeline.

8.3 Test Results

8.3.1 Consistency Sampling and Assaying

Oil sand feed entering the ATP processor and tailings exiting it were sampled at 30-minute intervals and assayed for consistency. Oil sand samples two kilograms in size were grabbed from the feed conveyor, while tailings were obtained by filling a 20-litre pail, keeping it mixed, and then sub-sampling it to produce 13 two-kilogram samples.

Oil/water/solids assays of the oil sand samples were performed using the Dean-Stark extraction method. Extracted solids were reserved for determination of particle size distributions (PSD). Water in tailings samples was determined by oven drying at 110°C, while hydrocarbon content was obtained as part of the chemical analysis.

Oil sand assays averaged 10.6% oil, 8.0% water, and 81.4% solids by weight. Eight percent water is slightly high for oil sand; this is attributable to rainfall. Tailings solids contained no hydrocarbon but did contain from 8.3 to 21.4% by weight of water. This variation is congruent with variations in tailings output and quench water spray rates.

Sieve and hydrometer tests were used to determine PSDs. For both extracted oil sand solids and tailings solids, PSDs lay within a fairly narrow band and the average PSD of each stream is representative of fine to medium sand as shown in Table 8-1. Tailings have significantly smaller <44, <22, and <2 μm size fractions which could be indicative of agglomeration of clay particles with one another or with silt and sand particles. This shift is illustrated graphically in Figure 8-2.

TABLE 8-1. Particle size distributions.

Wt% oil sand particle	Size range	Wt% tailings particles	Size range
32	<80 μm	25	<80 μm
21	<44 μm	11	<44 μm
16	<22 μm	8	<22 μm
5	<2 μm	3	<2 μm

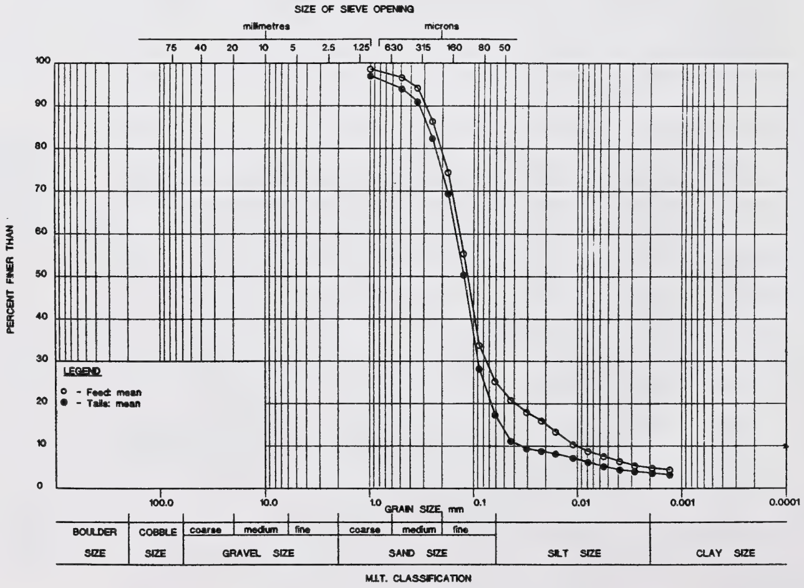


FIGURE 8-2. Comparison of grain size distribution for oil sand feed and tailings materials.

8.3.2 Baseline Index Tests

A series of tests was carried out on tailings and oil sand solids to provide indications of the content and activity of clays and other sludge forming constituents. It was hoped that the results would be useful in assessing changes in index properties resulting from the ATP process and in assessing the potential for sludge formation during hydraulic deposition.

Methylene Blue Index — The methylene blue index of tailings is 83 mL/100 g as compared to 183 mL/100 g for extracted oil sand solids. These results reflect a reduced cation exchange capacity for tailings which is indicative of a reduced clay content and/or an alteration in the clay minerals.

Specific Surface Area Test — Tailings solids have a specific surface area of 15.9 m^2/g as opposed to 22.3 m^2/g for oil sand solids. This is indicative of the change in PSD, for sure, and possibly of an alteration of particle shape. A rounding of flat, plate-like particles could also reduce the specific surface area of tailings solids.

Amorphous Iron — Results of this test indicate that the ATP does not significantly alter Fe_2O_3 since it is present in both tailings and feed to the extent of 0.37 percent by weight.

X-ray Diffraction (XRD) — The <80 μm fractions of tailings solids and extracted oil sand solids were submitted for XRD analysis. Both kaolinite and illite clay minerals were detected in the feed, whereas only illite was detected in tailings indicating alteration of kaolinite through heating.

Scanning Electron Microscopy (SEM) — Examination of samples by SEM shows that the majority of clay-shaped particles present in the feed are absent from tailings, even though X-ray elemental spectroscopy reveals no alteration in the elemental content of tailings. This is consistent with standard thermal analysis for kaolinite which indicates that kaolinite minerals are irreversibly altered to metakaolin by heating them to 500° to 550°C.

8.3.3 Engineering Properties of Slurried Tailings

To assess the potential for pipelined tailings to segregate during deposition and to investigate the strength characteristics of separated fine solids we conducted sedimentation column tests, consolidation tests, and direct shear tests on prepared fine solids specimens.

Standpipe Sedimentation — Three sedimentation tests were carried out on ATP tailings, the first two (Figures 8-3 and 8-4) on total tails adjusted to initial solids concentrations of 14 and 28 percent by weight, respectively. Within this range, solids content had only a minor effect on settling behavior. Distinct coarse and fine tailings phases formed readily with the division between phases occurring at the 80 μm particle size. Solids content at the bottom of the coarse phase was almost 85 percent. At the top of the fine tailings phase it was about 45 percent. Settling behavior in the standpipe displayed a relatively high maximum excess head within the first few minutes, dissipating to zero within hours.

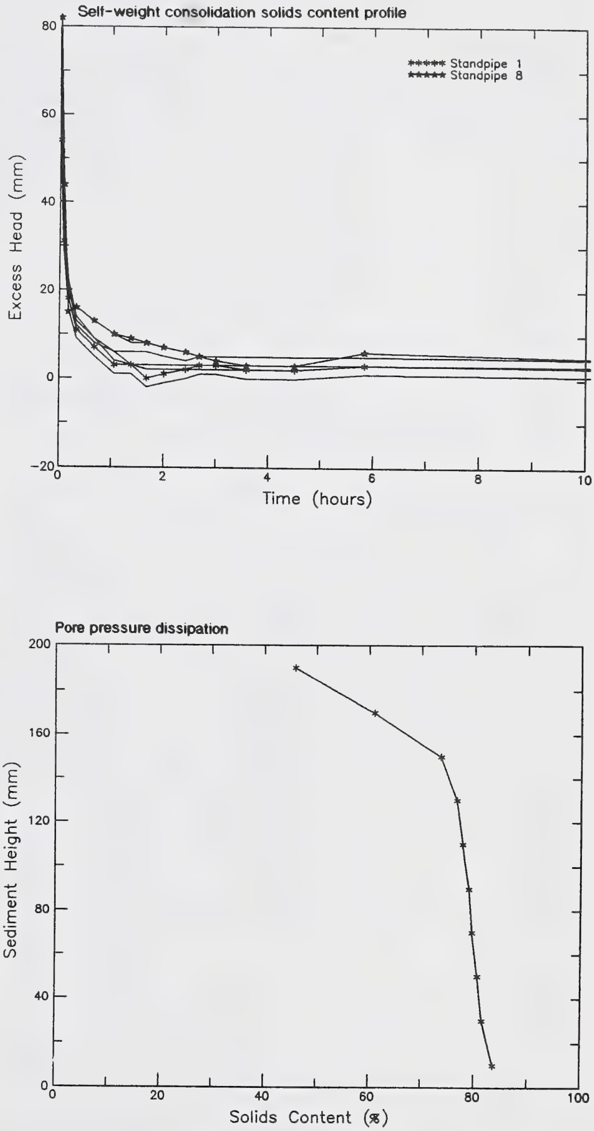


FIGURE 8-3. Summary of sedimentation column test results — total tailings (14 percent initial solids content).

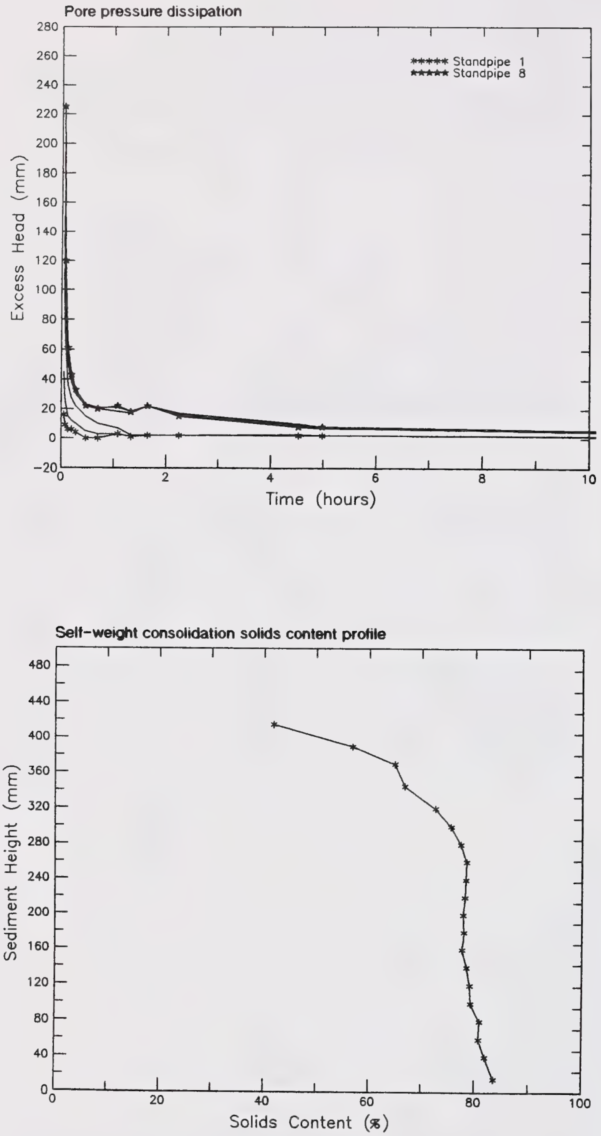


FIGURE 8-4. Summary of sedimentation column test results — total tailings (28 percent initial solids content).

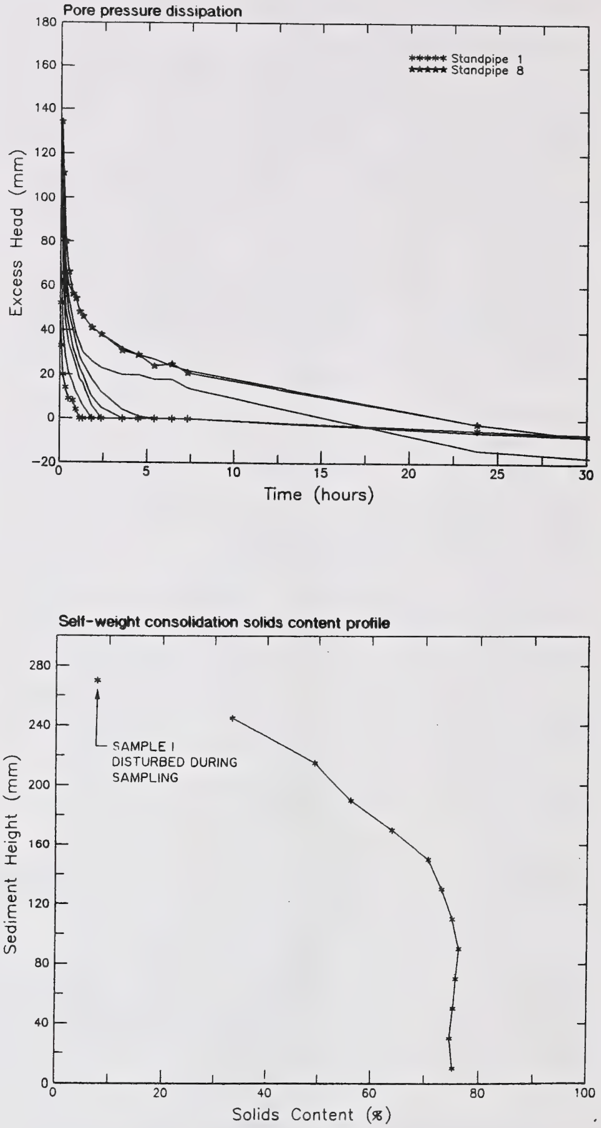


FIGURE 8-5. Summary of sedimentation column test results — minus 80 fraction (15 percent initial solids content).

The third test (Figure 8-5) was carried out using only the <80 μm fraction from total tailings. The initial settling concentration was adjusted to 15 percent solids by weight. As expected, it took slightly longer to attain sediment height stability and 24 hours was needed to reach zero excess head. The plot of solids content *versus* sediment height was also slightly lower but in a similar range.

Direct Shear Tests — Four direct shear tests were performed on drained samples of sediment from the third sedimentation column test, that is, on sediment composed entirely of <80 μm tailings solids. The results indicate an average peak friction angle of 35 degrees with no post peak strain softening. This angle is typical of a natural loose sand or compact silt deposit.

Consolidation Test — One saturated, one-dimensional oedometer consolidation test was performed on the <80 μm tailings fraction. The result was a compression index of 0.35. Consolidation occurred very quickly, indicating a very highly pervious material.

8.3.4 Environmental Impact Tests

Standard, screening level, laboratory toxicity tests, and chemical analyses were carried out on simulated leachate from ATP tailings. These tests included: Cereodaphnia 7-day survival and reproduction; algal growth inhibition/stimulation; flathead minnow survival and growth; and chemical analyses for specific cations, anions, hydrocarbons, and phenols.

Toxicity Tests — Screening level tests were conducted to give a preliminary indication of the potential environmental impact of ATP tailings and to provide data for comparison with tailings from other processes. The extract used for these tests was prepared by shaking one part of dry ATP tailings in four parts (by weight) of de-ionized water for two minutes then allowing them to stand overnight. Next day the extract was decanted from the sediment and introduced to colonies of four different organisms: cereodaphnia dubia, photobacteria phosphorem (Microtox screening assay), selenastrum capricornutum, and flathead minnow.

Cereodaphnia exhibited 100 percent mortality in the full strength extract. The estimated "no observed effect" concentration (NOEC) for this extract is 10 percent for mortality and one percent for reproduction. On this basis the extract was described as moderately toxic. Microtox results showed that the undiluted extract is not toxic to photobacterium phosphorem. The 72-hour algal growth inhibition tests indicated that the NOEC is 25 percent for selenastrum capricornutum. Finally, the flathead minnow larval growth and survival test indicated a NOEC for this extract of 10 percent for both mortality and fish growth.

These screening-level tests provide only preliminary indications of toxicity. They are not indicative of ultimate dilution levels in interactions with local soil and water systems for a commercial project.

Chemical Analyses — Chemical analyses were carried out on feed and tailings solids and their respective leachate extracts. The results (Appendix A) are intended to augment the chemistry database and to complement the toxicity data. The data show that the leachate extract surpasses drinking water requirements. However its ionic content is high, particularly in zinc, and this might explain the slight toxicity at the concentrations tested.

8.3.5 Engineering Properties of Dry Tailings

Tests that were performed on dry tailings to quantify engineering properties needed to design and operate a dry tailings disposal scheme included: California Bearing Ratio (CBR), monotonic triaxial liquefaction, Proctor Compaction, bulk density, relative density, and one-dimensional consolidation.

Bulk Density — Nine Shelby-tube samples obtained from the tailings pile just under the conveyor discharge were weighed and bulk densities calculated. The moisture content of the samples ranged from 9.4 to 11.7 percent by weight. This is at the low end of results obtained during consistency sampling. These results reveal a mean *in situ* dry density of 1224 kg/m^3 which is very loose.

Proctor Compaction — One Standard Proctor Compaction test (ASTM D-698) was carried out on bulk tailings to determine the moisture-density relationship. There was a well defined peak with a maximum dry density of 1660 kg/m^3 at an optimum moisture content of 15.5 percent. This is typical of a narrowly graded sand.

Relative Density — One relative density determination (ASTM D-4253) was carried out on bulk tailings, and minimum and maximum dry densities of 1397 kg/m^3 and 1761 kg/m^3 were obtained. The lower density observed in the Shelby-tube samples is likely due to partial saturation of the tailings which leads to very loose packing.

California Bearing Ratio — A single-point CBR test was carried out (in accordance with ASTM) on a bulk sample of tailings (soaked and unsoaked) compacted to approximately 100 percent maximum dry density as determined by Standard Proctor test. The result was a CBR of 10 percent for unsoaked or soaked tailings.

Consolidation Tests — Three one-dimensional oedometer consolidation tests were performed on a bulk sample of tailings in accordance with ASTM D-2435. In the first test the loosely placed sample was loaded, in its moist state, to a vertical stress of 2,500 kPa which is equivalent to a dry dump, burial depth of approximately 150 m. The second and third tests were completed in the same manner, except that the samples were inundated with water at vertical stresses of 20 and 270 kPa, respectively, to simulate saturation of a deposit at various stages of tailings dump development.

Based on the results summarized in Figure 8-6, the average compression index defined by $Cc = [e_0 - e_1]/\log[\sigma_1'/\sigma_0']$, is 0.23 for a vertical load range of 80 to 2,000 kPa. Some collapse of the tailings is evident when it is inundated with water. In all tests, primary compression of the 10 mm thick sample was completed within 15 seconds of applying load. This rapid compression is indicative of a highly pervious material.

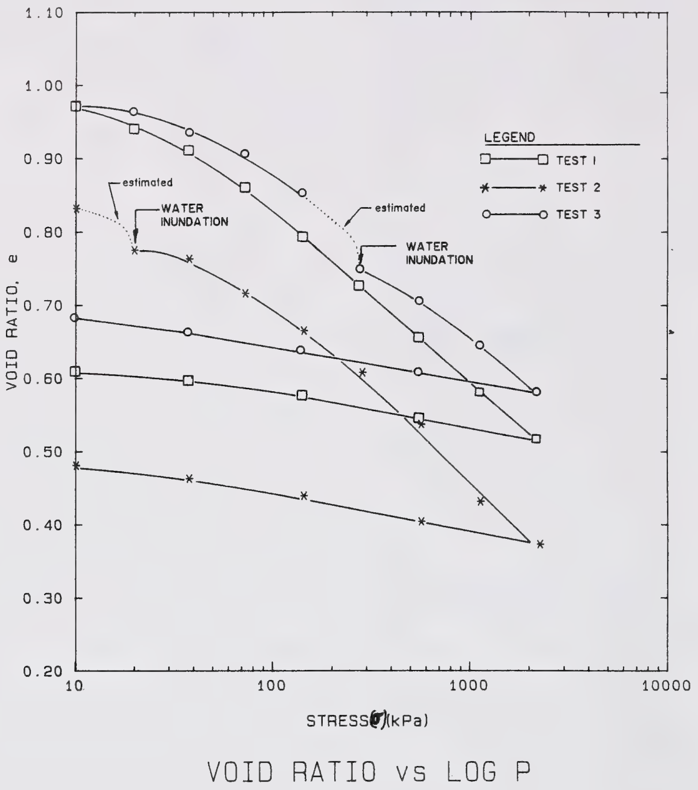


FIGURE 8-6. One-dimensional consolidation test results.

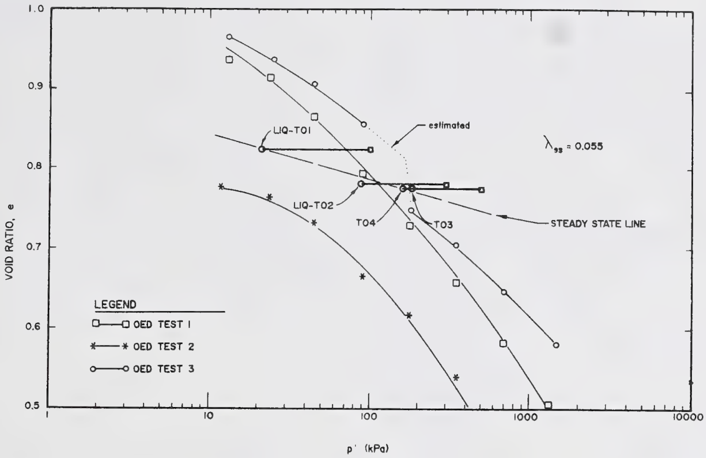
Liquefaction Tests — Four stress-controlled, consolidated, undrained triaxial tests were carried out on a bulk sample of ATP tailings, and the results are summarized in Figure 8-7 along with the equations used to convert oedometer vertical load to mean stress. The results show that dry-placed tailings have a high liquefaction potential at low confining stresses, that is, low burial depths. The liquefaction potential is reduced at higher confining pressures. Note that "dry-placed" tailings would have a quench water moisture content of about 10 percent and that this would result in a partially saturated deposit that could be expected to be very loose due to surface tension effects.

8.4 Discussion

Results of comparative testing show that tailings mineral soil has a lower fines content than the feed mineral soil. This is particularly evident in PSD results and is supported by the reduced methylene blue index and reduced specific surface area results. Scanning electron micrographs reveal clay mats in feed material but not in tailings. Further, X-ray diffraction tests indicate that the feed material contains up to 14 percent kaolinite while the tailings contain none. Both feed and tailings were estimated to contain 2.5 percent illite. X-ray diffraction results, which are semi-quantitative, indicate a higher clay fraction in tailings than do PSD results.

It is evident that temperatures in the Taciuk Process are sufficient to irreversibly alter kaolinite to metakaolin which is relatively non-colloidal and behaves in a particulate manner. Grain size distribution results suggest that at least a portion of the metakaolin particles aggregate together or attach themselves to silt and sand size particles. SEM results showed a reduction in platey clay particles, but could not confirm particle conglomeration.

Illite loses most of the water in its internal lattice at temperatures between 500° and 650°C. However, the lattice is not destroyed and irreversible alteration does not occur until



NOTE:

OED RESULTS CONVERTED VERTICAL LOAD TO p' USING:

$p' = (\sigma_1 + 2\sigma_3)/2$ $\frac{\sigma_3}{\sigma_1} = ko$ $ko = 1 - \sin\phi$

$\sin\phi = 35°$

FIGURE 8-7. Static liquefaction test results.

900°C. Thus illite tends to rehydrate despite being heated to 750°C in the ATP. Consequently, it is not surprising that kaolinite is eliminated from ATP tailings while illite persists.

Sedimentation and consolidation test results support the notion that ATP tailings are non-colloidal in character. The behavior of slurried tailings after they are discharged into a tailings pond can be imagined as follows. Initially, the slurry contains mineral particles ranging in sized from sand down to clay. These particles are suspended in water and do not interact with each other, therefore, there is no effective stress within the slurry.

Sand sized particles quickly drop out of suspension to form a loose soil in which particles are in physical contact and inter-granular voids are filled with water (pore water). Stresses imposed on this sediment, such as those from the weight of further sediment, are resisted partly by grain-to-grain physical contact and partly by the induced hydraulic pressure of the pore water. Accordingly, there is an effective stress in the soil that increases as excess pore water pressure dissipates with drainage. This process is called “consolidation.”

Silt and clay sized particles tend to remain suspended longer because their lower mass-to-surface area ratios render them more susceptible to inter-particulate electrical and chemical interactions than to gravity. In the AOSTRA Taciuk Process the elimination of the majority of colloidal particles from tailings significantly reduces their propensity to form sludge. Indeed, because they behave in a particulate rather than a colloidal manner their settlement and consolidation properties are dominated by particle-to-particle physical contact rather than by electrical forces.

On the other hand, in suspensions resulting from conventional water-based extraction processes the fine tails undergo “hindered settling” for years or decades during which time the particles remain supported by the fluid and are kept apart by strong particle-to-particle interactions which lead to the formation of a gel. Such interactions are generally associated with charge deficiencies on the surfaces of clay particles that result in a bound layer of oriented water molecules around each particle. This layer effectively increases the size of each particle and sets up repulsive forces between particles. Since the particles are supported by the fluid, there is no effective stress in the soil and further loading of the material is ineffective in initiating consolidation.

Because ATP tailings consolidate rapidly and because there is a large effective stress in the deposit, a beaching type of operation is feasible with complete and immediate water recycle. This disposal technique should result in a much smaller tailings dump footprint and the ability to produce a dry, final landscape.

Results from preliminary toxicity screening and chemical analysis suggest that leachate from an ATP tailings deposit would have a minor toxic effect, related possibly to an ionic imbalance. In a commercial operation, interactions with natural water and soil systems should have an ameliorative effect on such an imbalance. Additional toxicity testing, chemical analyses and simulations of soil/water interactions are required to arrive at a realistic evaluation of the toxicity potential of an operating plant.

For "dry" tailings disposal schemes, the potential for static liquefaction failure of tailings deposits must be considered. The surface of a dry-placed deposit would not be trafficable by heavy vehicles. As a preliminary guideline, long-term haul roads for large trucks will likely require a 2 m sub-base of compacted tailings overlain with a 1 m base course of compacted, crushed gravel. Maintenance requirements will have to consider the potential for collapse and differential settlements due to wetting.

8.5 Conclusions

- Wet disposal of ATP tailings should be considered the preferred option. Significant alteration of clays at process temperatures results in tailings with high permeability that consolidate rapidly and have no apparent potential for sludge formation.
- Dry disposal of ATP tailings requires a design that mitigates against the potential for liquefaction, if there is any chance of sloped deposits becoming saturated. Special haul roads will be required for heavy vehicles operating on a dry disposed tailings deposit.
- A dry, final landscape should be compatible with hydraulic transportation of ATP tailings.

8.6 Recommendations

- A large-scale field test is needed to confirm the conclusion about a dry, final landscape.
- A more detailed evaluation of leachate toxicity potential is also recommended. Further studies should consider the perspective of an operating plant and its interaction with natural surface drainage, ground water and soil systems.

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GOLDER ASSOCIATES
ATTENTION : DAVE HORSFIELD

Sample Description : SAMPLE # 1 FEED
Sample Date & Time : 06-10-92 REC'D
Sampled By : D.H.
Sample Type : COMPOSITE
Sample Station Code :

Chemex Worksheet Number : 92-02704-1
Chemex Project Number : GOLD121-0501
Sample Access :
Sample Matrix : SOIL
Report Date : October 29, 1992

PARAMETER DESCRIPTION	NAQUADAT CODE	UNITS		RESULTS	DETECTION LIMIT
Extraction Ratio		: 1		10.	1.
Soluble Chloride		mg/L		2.5	0.1
Total Sulphur		wt. %		0.14	0.01
Bromide - Water Soluble		ug/g	<	0.02	0.02
Hexavalent Chromium		ug/g		0.060	0.003
Fluoride (Water Soluble)		ug/g		0.48	0.05
Phenolics		ug/g		0.28	0.01
Arsenic (AA)		ug/g		0.3	0.2
Selenium (AA)		ug/g	<	0.2	0.2
Antimony (AA)		ug/g	<	0.2	0.2
Total Mercury- (CVAA)		ug/kg		47.	20.
Aluminum (ICP)		ug/g		1060.	1.
Barium (ICP)		ug/g		27.0	0.1
Beryllium (ICP)		ug/g		0.3	0.1
Boron (ICP)		ug/g		5.	1.
Cadmium (ICP)		ug/g	<	0.3	0.3
Calcium (ICP)		ug/g		1510.	20.
Chromium (ICP)		ug/g		4.0	0.2
Cobalt (ICP)		ug/g		2.4	0.1
Copper (ICP)		ug/g		2.1	0.2
Iron (ICP)		ug/g		3290.	1.
Lead (ICP)		ug/g	<	2.	2.
Lithium (ICP)		ug/g		1.7	0.5
Magnesium (ICP)		ug/g		520.	20.
Manganese (ICP)		ug/g		69.9	0.1
Molybdenum (ICP)		ug/g		0.6	0.3
Nickel (ICP)		ug/g		6.	1.
Phosphorous (ICP)		ug/g		40.	10.
Potassium (ICP)		ug/g		250.	20.
Silicon (ICP)		ug/g		579.	5.
Silver (ICP)		ug/g		0.5	0.2
Sodium (ICP)		ug/g		110.	20.
Strontium (ICP)		ug/g		8.4	0.5
Titanium (ICP)		ug/g		49.3	0.5

Reviewed By:

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GOLDER ASSOCIATES
ATTENTION : DAVE HORSFIELD

Sample Description : SAMPLE # 1 FEEO
Sample Date & Time : 06-10-92 REC'D
Sampled By : D.H.
Sample Type : COMPOSITE
Sample Station Code :

Chemex Worksheet Number : 92-02704-1
Chemex Project Number : GOLD121-0501
Sample Access :
Sample Matrix : SOIL
Report Date : October 29, 1992

PARAMETER DESCRIPTION	NAQUADAT CODE	UNITS	RESULTS	DETECTION LIMIT
Uranium (ICP)		ug/g	< 50.	50.
Vanadium (ICP)		ug/g	5.7	0.2
Zinc (ICP)		ug/g	9.5	0.1

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GOLDER ASSOCIATES
ATTENTION : DAVE HORSFIELD

Sample Description : TAILINGS BULK B1
Sample Date & Time : 15-09-92 REC'D
Sampled By : D.H.
Sample Type : COMPOSITE
Sample Station Code : JOB# 922-2403

Chemex Worksheet Number : 92-02422-1
Chemex Project Number : GOLD121-0501
Sample Access :
Sample Matrix : SOIL
Report Date : September 28, 1992

PARAMETER DESCRIPTION	NAQUADAT CODE	UNITS	R E S U L T S	DETECTION LIMIT
Extraction Ratio		: 1	2.	1.
Soluble Chloride		mg/L	2.3	0.1
Total Sulphur		wt. %	< 0.1	0.1
Bromide - Water Soluble		ug/g	< 0.02	0.02
Hexavalent Chromium		ug/g	< 0.02	0.02
Fluoride (Water Soluble)		ug/g	0.57	0.05
Phenolics		ug/g	< 0.01	0.01
Arsenic (AA)		ug/g	0.4	0.2
Selenium (AA)		ug/g	< 0.2	0.2
Antimony (AA)		ug/g	< 0.2	0.2
Total Mercury- (CVAA)		ug/kg	< 20.	20.
Aluminum (ICP)		ug/g	7930.	1.
Barium (ICP)		ug/g	54.0	0.1
Beryllium (ICP)		ug/g	0.3	0.1
Boron (ICP)		ug/g	3.	1.
Cadmium (ICP)		ug/g	0.5	0.3
Calcium (ICP)		ug/g	2880.	20.
Chromium (ICP)		ug/g	8.4	0.2
Cobalt (ICP)		ug/g	2.7	0.1
Copper (ICP)		ug/g	1.1	0.2
Iron (ICP)		ug/g	4050.	1.
Lead (ICP)		ug/g	< 2.	2.
Lithium (ICP)		ug/g	4.6	0.5
Magnesium (ICP)		ug/g	1180.	20.
Manganese (ICP)		ug/g	93.8	0.1
Molybdenum (ICP)		ug/g	0.3	0.3
Nickel (ICP)		ug/g	12.	1.
Phosphorous (ICP)		ug/g	72.	10.
Potassium (ICP)		ug/g	1140.	20.
Silicon (ICP)		ug/g	240.	5.
Silver (ICP)		ug/g	< 0.2	0.2
Sodium (ICP)		ug/g	102.	20.
Strontium (ICP)		ug/g	16.6	0.5
Titanium (ICP)		ug/g	52.3	0.5

Reviewed By:

GOLDER ASSOCIATES
ATTENTION : DAVE HORSFIELD

Sample Description : TAILINGS BULK B1
Sample Date & Time : 15-09-92 REC'D
Sampled By : D.H.
Sample Type : COMPOSITE
Sample Station Code : JOB# 922-2403

Chemex Worksheet Number : 92-02422-1
Chemex Project Number : GOLD121-0501
Sample Access :
Sample Matrix : SOIL
Report Date : September 28, 1992

PARAMETER DESCRIPTION	NAQUADAT CODE	UNITS	RESULTS		DETECTION LIMIT
Uranium (ICP)		ug/g	<	50.	50.
Vanadium (ICP)		ug/g		24.3	0.2
Zinc (ICP)		ug/g		18.4	0.1

Reviewed By:

COMPANY :GOLDER ASSOCIATES LTD.
ATTENTION :DAVE HORSFIELD
Client Job# :922-2403
Sample ID# :
Matrix :SOLID TAILINGS B1
Chemex ID# :92-02422-01

Date Sampled :SEP 11,92
Date Received :SEP 15,92
Date Analyzed :SEP 21,92

TOTAL EXTRACTABLE HYDROCARBONS

COMPONENT	BOILING RANGE CELCIUS	WEIGHT ppm
C7	68.8 TO 98.4	ND
C8	98.5 TO 125.7	ND
C9	125.8 TO 150.8	ND
C10	150.9 TO 174.2	ND
C11	174.3 TO 196.0	ND
C12	196.1 TO 216.0	ND
C13	216.1 TO 236.0	ND
C14	236.1 TO 253.0	ND
C15	253.1 TO 271.0	ND
C16	271.1 TO 287.0	ND
C17	287.1 TO 302.0	ND
C18	302.1 TO 317.0	ND
C19	317.1 TO 331.0	ND
C20	331.1 TO 344.0	ND
C21	344.1 TO 357.0	ND
C22	357.1 TO 369.0	ND
C23	369.1 TO 380.0	ND
C24	380.1 TO 391.0	ND
C25	391.1 TO 402.0	ND
C26	402.1 TO 412.0	ND
C27	412.1 TO 422.0	ND
C28	422.1 TO 432.0	ND
C29	432.1 TO 441.0	ND
C30	441.1 TO 449.0	ND
TOTAL		ND

NA -	Not analyzed.
ND -	Not detected or below detectable limit.
TRACE -	Components above method detection limit but below practical quantitaion limit.

AVERAGE MOLECULAR WEIGHT : 0 AMU

QA/QC SUMMARY

SURROGATE RECOVERY : 103 %
LIQUID SURROGATE REC. LIMITS : 20 % TO 75 %
SOLID SURROGATE REC. LIMITS : 81 % TO 119 %
METHOD DETECTION LIMIT : 1.0 ppm PER COMPONENT
PRACTICAL QUANTITATION LIMIT : 2.9 ppm PER COMPONENT

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GOLDER ASSOCIATES
ATTENTION : DAVE HORSFIELD

Sample Description : SAMPLE # 1 FEED
Sample Date & Time : 06-10-92 REC'D
Sampled By : D.H.
Sample Type : COMPOSITE
Sample Station Code :

Chemex Worksheet Number : 92-02704-1
Chemex Project Number : GOLD121-0501
Sample Access :
Sample Matrix : ~~SOIL~~ LEACHAT
Report Date : October 29, 1992

PARAMETER DESCRIPTION	NAQUADAT CODE	UNITS		RESULTS	DETECTION LIMIT
Calcium - (ICP) Extractable	20311L	mg/L		65.80	0.01
Magnesium - (ICP) Extractable	12311L	mg/L		9.80	0.01
Sodium - (ICP) Extractable	11311L	mg/L		4.80	0.01
Potassium -(ICP) Extractable		mg/L		1.50	0.02
Chloride - (IC)	17209L	mg/L		1.20	0.01
Silicon - Extractable (ICP)		mg/L		2.73	0.02
Fluoride	09105L	mg/L		1.39	0.05
Phenols	06537L	mg/L		0.014	0.001
Bromide (IC)		mg/L	<	0.1	0.1
Sulphur - (ICP) - Extractable		mg/L		7.9	0.2
Aluminum - Extractable (ICP)	13311L	mg/L		0.80	0.01
Antimony - Extractable (AA)	51003L	mg/L	<	0.002	0.002
Arsenic - Extractable (AA)	33005L	mg/L		0.004	0.002
Barium - Extractable (ICP)	56311L	mg/L		0.17	0.01
Beryllium - Extractable (ICP)	04311L	mg/L		0.001	0.001
Boron - Extractable (ICP)	05211L	mg/L		0.60	0.01
Cadmium - Extractable (ICP)	48501L	mg/L	<	0.003	0.003
Chromium - Extractable (ICP)	24360L	mg/L		0.002	0.002
Cobalt - Extractable (ICP)	27360L	mg/L		0.029	0.003
Copper - Extractable (ICP)	29501L	mg/L		0.001	0.001
Iron - Extractable (ICP)	26311L	mg/L		0.83	0.01
Lead - Extractable (ICP)	82311L	mg/L	<	0.02	0.02
Lithium - Extractable (ICP)	03309L	mg/L		0.007	0.001
Manganese - Extractable (ICP)	25311L	mg/L		1.240	0.001
Mercury - Extractable (CVAA)	80301L	ug/L	<	0.05	0.05
Molybdenum - Extractable (ICP)	42330L	mg/L	<	0.003	0.003
Nickel - Extractable (ICP)	28350L	mg/L		0.062	0.005
Phosphorous - Extractable (ICP)	15450D	mg/L	<	0.05	0.05
Selenium - Extractable (AA)	34005L	mg/L	<	0.002	0.002
Silver - Extractable (ICP)	47311L	mg/L	<	0.001	0.001
Strontium - Extractable (ICP)	38311L	mg/L		0.150	0.002
Titanium - Extractable (ICP)		mg/L	<	0.002	0.002
Uranium - Extractable (ICP)		mg/L	<	0.5	0.5
Vanadium - Extractable (ICP)	23311L	mg/L		0.002	0.002

NOTES : The above extraction was conducted according to the procedure described in the General Standards Board Class 9, Miscellaneous Substances: CGSB Leachate Extraction Procedure:. Analysis methods used were as set out in the US EPA SW-846 "Test Methods for Evaluating Solid Waste".

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GOLDER ASSOCIATES
ATTENTION : DAVE HORSFIELD

Sample Description : SAMPLE # 1 FEED
Sample Date & Time : 06-10-92 REC'D
Sampled By : D.H.
Sample Type : COMPOSITE
Sample Station Code :

Chemex Worksheet Number : 92-02704-1
Chemex Project Number : GOLD121-0501
Sample Access :
Sample Matrix : ~~SOI~~L LEACHATE
Report Date : October 29, 1992

PARAMETER DESCRIPTION	NAQUADAT CODE	UNITS	RESULTS	DETECTION LIMIT
Zinc - Extractable (ICP)	30501L	mg/L	0.037	0.001

Reviewed By:

CHEMEX Labs Alberta Inc.

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GOLDER ASSOCIATES
ATTENTION : DAVE HORSFIELD

Sample Description : TAILINGS BULK B1
Sample Date & Time : 15-09-92 REC'D
Sampled By : D.H.
Sample Type : COMPOSITE
Sample Station Code : JOB # 922-2403

Chemex Worksheet Number : 92-02422-1L
Chemex Project Number : GOLD121-0501
Sample Access :
Sample Matrix : LEACHATE
Report Date : October 8, 1992

PARAMETER DESCRIPTION	NAQUADAT CODE	UNITS	RESULTS	DETECTION LIMIT
Calcium - (ICP) Extractable	20311L	mg/L	163.00	0.01
Magnesium - (ICP) Extractable	12311L	mg/L	32.60	0.01
Sodium - (ICP) Extractable	11311L	mg/L	1.10	0.01
Potassium -(ICP) Extractable		mg/L	3.60	0.02
Silicon - Extractable (ICP)		mg/L	8.46	0.02
Sulphur - (ICP) - Extractable		mg/L	55.0	0.2
Aluminum - Extractable (ICP)	13311L	mg/L	0.58	0.01
Barium - Extractable (ICP)	56311L	mg/L	0.09	0.01
Beryllium - Extractable (ICP)	04311L	mg/L	0.002	0.001
Boron - Extractable (ICP)	05211L	mg/L	0.13	0.01
Cadmium - Extractable (ICP)	48501L	mg/L	0.011	0.003
Chromium - Extractable (ICP)	24360L	mg/L	0.009	0.002
Cobalt - Extractable (ICP)	27360L	mg/L	0.005	0.003
Copper - Extractable (ICP)	29501L	mg/L	0.002	0.001
Iron - Extractable (ICP)	26311L	mg/L	0.08	0.01
Lead - Extractable (ICP)	82311L	mg/L	< 0.02	0.02
Lithium - Extractable (ICP)	03309L	mg/L	0.022	0.001
Manganese - Extractable (ICP)	25311L	mg/L	1.150	0.001
Molybdenum - Extractable (ICP)	42330L	mg/L	0.012	0.001
Nickel - Extractable (ICP)	28350L	mg/L	0.031	0.005
Phosphorous - Extractable (ICP)	15450D	mg/L	0.2	0.1
Silver - Extractable (ICP)	47311L	mg/L	< 0.002	0.002
Strontium - Extractable (ICP)	38311L	mg/L	0.450	0.002
Titanium - Extractable (ICP)		mg/L	< 0.003	0.003
Uranium - Extractable (ICP)		mg/L	< 0.5	0.5
Vanadium - Extractable (ICP)	23311L	mg/L	0.022	0.002
Zinc - Extractable (ICP)	30501L	mg/L	0.065	0.001

Note: The above extraction was conducted according to the procedure described in "The Canadian General Standards Board Class 9, Miscellaneous Substances: CGSB Leachate Extraction Procedure". Analysis methods used were as set out in the US EPA SW-846 "Test Methods for Evaluating Solid Waste".

Reviewed By: ______________________

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